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ANALYTICAL ABSTRACTS

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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

2018. **Diallyldithiocarbamidohydrazine as an analytical reagent.** N. K. Dutta and K. P. Sen Sarma (*Science and Culture*, 1955, **20** [8], 397).—The reagent was used by Gupta and Sen Sarma (*Brit. Abstr. C*, 1952, 47) for the colorimetric determination of bismuth. It has now been observed that the reagent precipitates Cu and Ni quant. at pH 2·5 to 3·5 and 8·1 to 8·7, respectively. The reagent has also been used for the determination of these metals in a mixture by pptg. successively at these pH values. The nickel compound contains 19·8 per cent. of Ni and can be weighed after washing with acetone. The copper compound must be washed with water, ignited to CuO, dissolved in nitric acid and determined iodimetrically. N. E.

2019. **A note on chloramine B as a volumetric reagent.** A. Singh (*J. Indian Chem. Soc.*, 1954, **31** [8], 647).—Chloramine B is recommended as a volumetric oxidising agent. It is readily soluble in water and the aqueous solution behaves as a hypochlorite, but is more stable than sodium hypochlorite. Arsenous oxide, tartar emetic, potassium thiocyanate, hydrazine sulphate, stannous chloride, ferrous sulphate, mercurous chloride and thallous chloride may be oxidised with iodine monochloride and the liberated iodine titrated with standard chloramine B. R. J. MAGEE

2020. **Studies on arsenic trichloride as a solvent. III. Potentiometric acid - base titrations in $AsCl_3$.** L. H. Anderson and I. Lindqvist (*Acta Chem. Scand.*, 1955, **9** [1], 79–83).—The possibilities of measuring large pCl changes in potentiometric acid - base titrations in $AsCl_3$ are dealt with. The definition acid + $Cl' \rightleftharpoons$ base leads to the use of the following concentration cell:
Ag, AgCl|Cl′(C_1)||Cl′(C_2)|Ag, AgCl. The acid $FeCl_3$ has been titrated against the bases tetramethylammonium chloride, pyridine and diethylamine, and the base tetramethylammonium chloride against the acids $FeCl_3$ and $SbCl_5$. A comparison with a conductometric titration has also been made.
C. H. WHITTON

2021. **Amperometric titrations with hypochlorite in the presence of bromide.** H. A. Laitinen and D. E. Woerner (*Anal. Chem.*, 1955, **27** [2], 215–217).—Arsenite, NH_3 and CNS′ are successfully determined amperometrically (rotating platinum electrode) with $Ca(OCl)_2$ in the presence of Br′. 0·1202 *N* As_2O_3 in $NaHCO_3$ (25 ml) is treated with 10 per cent. KBr - 5 per cent. $NaHCO_3$, and the soln. is titrated with 0·03785 *M* $Ca(OCl)_2$ at + 0·2 V *vs.* the S.C.E. CNS′ and NH_4^+ are titrated similarly. Titrations of urea, NO_2', SO_3'', S'', CN', S_2O_5'', PO_3'' and H_2O_2 are not successful. Results are presented for several concn. of the reducing agent and reagents. Sensitivity and accuracy are claimed to compare favourably with those of the direct titration.
D. A. PANTONY

2022. **Quinoline as a fluorescent indicator.** S. Šljivić, I. Burić and K. Nikolić (*Z. anal. Chem.*, 1955, **145** [1], 16–18).—Acid - base titrations are performed with the use of quinoline as a fluorescent indicator in u.v. light. The optimum bright-blue fluorescence (420 to 570 mμ) is given by concn. of 10^{-4} g per ml of quinoline in weakly acid solutions (pH 6·2 to 7·2). Mineral acids are titrated with NaOH, aq. NH_3 soln. and $Ba(OH)_2$, but HCl and HI at concn. > 0·1 *N* are not titratable, since the fluorescence is quenched. Organic acids are titrated with NaOH with the use of Polaroids instead of Nicol prisms to avoid loss of light intensity. Coloured (other than dark yellow) soln. are titratable, but not wine or acid solutions giving ppt. with quinoline. D. R. GLASSON

2023. **Theory and practice of chromatography.** R. A. Wells (*Pharm. J.*, 1955, **174** [4769], 242–244; [4770], 260–261).—A brief review is given of techniques used in chromatography, especially the use of surface-active adsorbents, partition chromatography on paper and on columns, gas - liquid systems, ion-exchange chromatography, and frontal analysis. S.C.I. ABSTR.

2024. **2:6-Dichlorophenolindophenol as a spray reagent [in chromatography].** J. Barnabas and G. V. Joshi (*Anal. Chem.*, 1955, **27** [3], 443–444).—2:6-Dichlorophenolindophenol can be used as a spray reagent in the paper chromatography of organic acids. Dark-pink spots are usually obtained immediately after spraying. Certain acids bleach the dye, thus aiding further differentiation.
G. P. COOK

2025. **A relation between R_F values of unidimensional and circular chromatography.** N. C. Ganguli (*Anal. Chim. Acta*, 1955, **12** [4], 335–341).—The R_F values of a number of amino acids and sugars are determined with the use of a phenol - water mixture and a benzyl alcohol - acetic acid - water mixture (100:20:26) as solvents in unidimensional and circular-paper chromatography. The respective R_F values for the unidimensional method are equal to the square of the R_F values for the circular chromatograms; the same conclusion is reached in a theoretical consideration.
W. C. JOHNSON

2026. **Ion exchangers (composition, properties and uses).** H. Deuel (*Mitt. Lebensmitt. Hyg., Bern*, 1955, **46** [1], 12–35).—Ion exchangers are reviewed under the following headings: (1) historical; (2) principles; (3) composition and synthesis of ion-exchange resins; (4) properties of ion exchangers, with special reference to their selectivity; (5) physical chemistry of ion-exchange reactions; (6) use in laboratory and industry. (164 references.)
K. J. GARDNER

2027. **Ion-exchange separations on chemically modified cellulose.** N. F. Kember and R. A. Wells (*Nature*, 1955, **175**, 512–513).—Separation of cation mixtures has been carried out on strips of paper

(20 cm × 1 cm) prepared from cotton fabric treated with urea phosphate. With 2 *N* NaCl as ascending developer, sharp bands of Fe^{+++}, Cu and Ni (≃ 50 μg each) were obtained; the R_F values were Fe^{+++} 0·0, Cu 0·3, Ni 0·5. Columns (6 cm × 1 cm) of phosphorylated paper pulp could also be used; these were converted into the sodium form, and a mixture of Fe^{+++}, Cu and Ni (≃ 5 mg each) was adsorbed and eluted with *M* $CaCl_2$. Separation of anion mixtures was attempted on sheets of paper prepared from cotton fabric treated with 2-aminoethyl hydrogen sulphate. Difficulties were experienced owing to the poor wetting properties of the sheets, but partial separation of Au, Pt, Pd and Rh as their chloro acids was achieved. A. R. ROGERS

2028. **Reduction in the error of spectrochemical analysis by the use of a weighed amount of the reference element.** G. Holdt and H. Schäfer (*Z. Naturforsch.*, 1954, **9b** [7], 506).—The experimental error in the quantitative spectral analysis of samples of niobium oxide (Nb_2O_5) in a d.c. arc was considerably reduced by the introduction of an empirically determined weighed quantity of the reference element (PbO). The mean error was reduced from 21 to 25 per cent. to 8 per cent. The variable error is due to variations in temperature of the arc and is not found in arrangements that use a controlled-arc circuit. E. KAWERAU

2029. **Identification of traces of elements by spectrography and paper chromatography.** M. E. Heros and L. M. Amy (*Bull. Soc. Chim. France*, 1955, [3], 367–369).—Paper chromatography is used first to separate elements that are normally not resolvable by the spectrograph; this is followed by spectrographic identification. At present, the method has been applied only qual., but it appears to be capable of being made quant. R. J. COLE

2030. **Differential measurements of reflectance [of cloth, etc.].** C. A. Lermond and L. B. Rogers (*Anal. Chem.*, 1955, **27** [3], 340–346).—Some reflectance data obtained spectrophotometrically are given for dyed cloth, powders and dyed yarns. The reproducibility is within 0·2 per cent. for the first two and within 2 per cent. for the last. More precise measurements at low reflectance can be obtained by the use of differential spectrophotometric measurements. Direct analyses of solid material by reflectance determinations appear to be possible; these should be particularly useful for difficultly soluble solid mixtures and for substances on chromatograms. J. H. WATON

2031. **Oscillographic polarography.** Ya. P. Gokhshteĭn and Yu. A. Surkov (*Zh. Anal. Khim., SSSR*, 1954, **9** [6], 319–343).—Methods of oscillographic polarography, including those of Matheson and Nichols, Randles, Delahay, Delahay as modified by the authors (*Anal. Abstr.*, 1954, **1**, 1746), Snowden and Page, Cruse and Heberle (*Z. Elektrochem.*, 1953, **57**, 579), Sevick and Heyrovsky, are reviewed and its advantages over ordinary polarography are discussed. Results of experiments with a saw-tooth wave are compared with theoretical results derived from the equations of Randles and of Sevick, and a semi-empirical equation is proposed to replace these equations. G. S. SMITH

2032. **Purification of supporting electrolytes for polarographic trace analysis by controlled potential electrolysis at mercury cathode.** L. Meites (*Anal. Chem.*, 1955, **27** [3], 416–417).—Material to be used as a supporting electrolyte in polarography can be freed from traces of heavy metals by electrolysis with a mercury cathode at suitable potentials. Details are given for the removal of Zn from NaOH, Ni and Zn from ammoniacal NH_4Cl, Fe from a citrate medium and alkali and alkaline-earth metals from tetramethylammonium hydroxide. The method is inapplicable to the removal of W, V and U. The elimination of Pb and Zn from reagents used in colorimetric estimations with dithizone might well be carried out by this method. J. H. WATON

2033. **Separation of substances by analytical and preparative distribution.** E. Hecker (*Öst. ChemZtg*, 1955, **56**, 3–11).—Compounds requiring special care (antibiotics, vitamins, fat-soluble azo dyestuffs, etc.) are best isolated by distribution processes. The principles of analytical (small-scale) and preparative (large-scale) distribution methods, based on concentrations of test substances, their specific distribution coeff. (*K*) and solubility in selected solvent-systems are explained. Discontinuous (Craig) and continuous (O'Keeffee; van Dyek) processes of multiple distribution are discussed in detail. The basic method of analytical multiple distribution consists in introducing one portion of the weighed test substance between known volumes of mobile upper and stationary lower phases of solvents (*e.g.*, water-saturated butanol and butanol-saturated water) and multiple transference of the upper fraction, after fresh additions of aq. solvent and establishment of equilibrium of the system. The relative amounts of substance contained in both solvent layers are determined and the weights of fractions plotted against the number of fractions. Suitable apparatus for both of the multiple distribution processes and their working techniques are described, *e.g.*, a Rometsch column, capable of separating two azo dyestuffs of $K = 0{\cdot}67$, $K_1 = 1{\cdot}6$ in a system containing *n*-heptanol - methanol. S.C.I. ABSTR.

2.—INORGANIC ANALYSIS

2034. **Spectrochemical procedure of general applicability.** E. K. Jaycox (*Anal. Chem.*, 1955, **27** [3], 347–350).—A semi-quant. method is described that will permit the determination of the metallic constituents of almost any material to ≃ ± 25 per cent., using a single set of standards. Copper oxide and graphite, as primary and secondary buffers, are added to the sample or standard diluted with GeO_2 in the ratio CuO: graphite: material + GeO_2 (19:20:1). In this way the matrix effect is kept at a min., the system approximating to that of a CuO - GeO_2 - graphite matrix. Spectra are recorded in the region 230 to 500 mμ. If the material for analysis is not a powder, it must first be converted into a salt or oxide. J. H. WATON

2035. **Paper chromatography of metal - 2-thenoyltrifluoroacetone chelates. I.** E. W. Berg and R. T. McIntyre (*Anal. Chem.*, 1954, **26** [5], 813–814).—Mixtures of chelates of 2-thenoyltrifluoroacetone with the following metals were completely separated on paper by a proper selection of the solvent system and adsorbent impregnated paper: Fe^{III}, Co^{II} and Ni^{II}; Fe^{III}, Ni^{II} and Mn^{II}; Cu^{II}, Ni^{II} and Mn^{II}; Cu^{II}, Ni^{II} and Co^{II}. Benzene - methanol (95:5) readily separated all except the last mixture on plain paper. Methanol - benzene - glacial acetic

acid (10:88:2) on alumina-impregnated paper was effective for all the mixtures. N. E.

2036. **Paper-chromatographic separation of metal - 2-thenoyltrifluoroacetone chelates. II.** E. W. Berg and R. T. McIntyre (*Anal. Chem.*, 1955, **27** [2], 195–198).—The complexes of 2-thenoyltrifluoroacetone with Fe^{III}, Cu^{II}, Ni^{II}, Co^{II} and Mn^{II} (*cf. Anal. Abstr.*, 1955, **2**, 2035) are purified by dissolution in ethanol and repptn. in water, and, after isolation and drying, they are dissolved in methyl *iso*propyl ketone. One-μl portions are spotted on filter-paper strip, and the dried spots are developed by upward-flow chromatography, with benzene - methanol - acetic acid as solvent. The complexes are located as follows: Fe and Cu by visual inspection, Mn with benzidine - NaOH reagent, Co with H_2S and Ni with dimethylglyoxime. R_F values fall in the order Fe, Cu > Ni > Co > Mn for all compositions of the developing solvent. Solubility, polarisation and polarisability are suggested as governing the relative strengths of adsorption of the chelates. D. A. Pantony

2037. **Complexiметric titrations (chelatometry). VIII. Screening of cations with 2:3-dimercaptopropanol.** R. Přibil and Z. Roubal (*Chem. Listy*, 1954, **48** [6], 818–824).—A 10 to 25 per cent. ethanolic soln. of 2:3-dimercaptopropanol (**I**) is an excellent screening agent for Pb, Bi and many other cations with which it forms colourless or faintly coloured salts soluble in aq. NH_3. Besides reacting with the free cations, **I** also reacts with their complexes with EDTA (**II**), quantitatively liberating **II**. Thus the use of **I**, either alone or in conjunction with other screening agents such as KCN and triethanolamine (**III**), results in a considerable increase in the selectivity of complexiметric determinations. *Determination of Mg in the absence of Ca, of Ca in the absence of Mg, or of total Ca plus Mg*—To the weakly acidic soln. add **I**, then a buffer soln. (prepared by mixing a soln. of 54 g of NH_4Cl in 300 ml of H_2O with 350 ml of 25 per cent. aq. NH_3 and diluting to 1 litre) until the ppt. dissolves, and titrate the mixture with 0·01 to 0·05 *M* **II**, using Eriochrome black T (**IV**) as indicator; at the end-point the colour changes from wine-red to clear blue. Pb, Cu, Cd and Hg do not interfere, nor does Bi in amounts $\not>$ 50 mg. *Determination of Ca in the presence of Pb, Mg, Fe, Al and other cations*—Treat the soln. with a few ml of **III** and then dropwise with **I** until the yellow ppt. of Pb or of Bi no longer forms; dissolve the ppt. by adding 10 to 20 ml of 2 *N* NaOH. Determine Ca by direct titration with **II** to murexide. Ni, Co, Mn and U give highly coloured complexes with **I** and must be absent. *Determination of Ni and Zn in the presence of each other*—Treat the soln. with an excess of **II**, basify it with the buffer soln. and determine the excess of **II** by titration with $MgSO_4$, with **IV** as indicator. This gives the total Ni and Zn content. Add a few ml of **I** (immediate colour change from wine-red to blue owing to the formation of Zn complex) and re-titrate the liberated **II** with $MgSO_4$, thus giving the Zn content. Proceed similarly to determine Ni in the presence of Hg, Cd, Pb or Bi. The procedure is unsuitable for determining Ni in the presence of Cu, Co or Mn. *Determination of Mn and Pb in the presence of each other*—To the weakly acidic soln. add 0·1 to 0·3 g of NH_2OH and a few ml of **III**, and determine the combined content of both metals by direct titration with **II**. To obtain the amount of Pb present, treat the soln. with a slight excess of **I** and titrate the liberated **II** with $MgSO_4$. *Determination of Ni, Zn and Mg (or total Ca plus Mg) in the same sample*—To the weakly acidic soln. add **II** in excess and determine the excess by titration with $MgSO_4$, giving combined Ni, Zn and Mg. After the addition of **I**, titrate the liberated **II**, equivalent to the Zn content, with $MgSO_4$. Finally add solid KCN to bind the Ni, and after 3 to 5 min. titrate the liberated **II**. *Determination of Pb, Co and Mn in the same sample*—Treat the weakly acidic soln. with NH_2OH, **III** and **II**, and by titrating with $MgSO_4$ determine the total content of the three metals. Now bind the Co with KCN and the Pb with **I**, titrating in each case the liberated **II** with $MgSO_4$. Any Al present is screened by **III** and does not interfere. The cation combinations Bi, Co and Mn, and Pb, Zn and Mg can be determined similarly. *Determination of Pb, Ni, Zn and Mg in the same sample*—Add to the soln. an excess of **II** and determine the excess with $MgSO_4$. Precipitate Pb with solid Na diethyldithiocarbamate and re-titrate the soln. with $MgSO_4$ to obtain the Pb content. Add **I** and titrate the liberated **II** equivalent to the Zn present. Finally bind Ni with KCN and re-titrate. The cation combinations of Cd, Zn, Ni and Mg, and Cd, Mn, Zn and Mg can be determined similarly. G. Glaser

2038. **Use of electrolytic apparatus for identification of alloys.** C. Goldberg (*Metallurgia*, 1955, **51** [305], 160).—Procedures are outlined briefly for the following approximate determinations: Zn in Al alloys; Mn in manganese bronze or Mn - Cu; Pb in brass, bronze, Babbitt metal and Zn; Fe in Fe - Cu mixtures; traces of Cu in Ni; and for rapidly removing small amounts of Cu or Cu and Pb before testing for other elements. G. C. Jones

2039. **Flame photometry. Quenching effect of chlorohydrocarbons on sodium and potassium estimations.** R. F. Milton and W. D. Duffield (*Chem. & Ind.*, 1955, [11], 280).—In the determination of Na and K by flame photometry considerable quenching occurs if organic halogen compounds, *e.g.*, chloroform, carbon tetrachloride and ethylene dichloride, are present in the solutions being analysed. D. R. Peck

2040. **Tetraethylthiuram disulphide as an analytical reagent. I. A new specific reaction for copper.** J. Michal and J. Zýka (*Chem. Listy*, 1954, **48** [6], 915–916).—A new specific reagent for the detection of Cu has been found in tetraethylthiuram disulphide (**I**). When a drop of a neutral or acid soln. containing Cu is treated with 2 to 3 drops of a saturated soln. of **I** in 96 per cent. ethanol, a yellow-brown coloration develops. Selenium and Hg^{+} interfere; the former can be masked with EDTA and in the presence of the latter a special procedure must be used. G. Glaser

2041. **Copper (I) - 2:2′-diquinolyl complex in aqueous dimethylformamide.** R. T. Pflaum, A. I. Popov and N. C. Goodspeed (*Anal. Chem.*, 1955, **27** [2], 253–255).—The solubility and light-absorption properties of the 2:2′-diquinolyl reagent in various solvents, effect of solvent and concn. of reagent and of Cu^{+}, of pH and of various other ions on the colour of the Cu^{I} - 2:2′-diquinolyl complex are investigated. The following method of determination of Cu^{II} is suggested: the sample (0·5 g) is treated with 6 *N* HCl (20 ml) and the soln. is taken to fumes with HNO_3 (5 ml) and H_2SO_4 (5 ml). After cooling, the residue is dissolved in hot water (30 ml) and the soln. is filtered. The filtrate and water washings are treated with tartaric acid (1 g)

and dil. NaOH to adjust the pH to 5 to 6, and the soln. is diluted to a suitable vol. with water. A 5-ml aliquot is added to the reagent [10^{-3} *M* 2:2′-diquinolyl in dimethylformamide (12·5 ml)] and the soln. is made up to 25 ml with water (the solvent reduces the Cu^{++} to Cu^{+}). The absorption of the purple soln. is measured at 545 mμ with respect to a solvent blank. Copper concn. is obtained from a standard calibration curve. Effects of 20 extraneous ions are listed; of these, Ag^{+}, Fe^{+++} and Pb^{++} interfere seriously. D. A. PANTONY

2042. **Method used in the Murex Laboratories for the determination of copper in refined copper.** E. A. Chidley (*Murex Rev.*, 1955, **1** [15], 424–425).—The assay solution is made by adding to 1100 ml of H_2O, first 315 ml of HNO_3 (sp. gr. 1·42) and then 450 ml of H_2SO_4 (sp. gr. 1·84), and cooling. *Procedure*—To 10·000 to 10·005 g of accurately weighed drillings, 80 ml of assay solution are added. After warming until the action has nearly ceased, solution is completed on a steam-bath at 80° to 90° C. After cooling, electrolysis is effected with a cylindrical perforated type of platinum cathode that weighs 20 to 30 g, and an anode of 17 S.W.G. platinum wire spiralled for about 2 in. at the bottom. A current of 1 amp. at 6 V is passed through the solution for approx. 16 hr. until electrolysis is completed. The cathode is washed, dried and weighed. Details and precautions are given.
C. H. WHITTON

2043. **The formation of complex ions used in analytical chemistry. IX. Studies on the complexes of copper, zinc and cadmium cyanides.** S. Suzuki (*J. Chem. Soc. Japan, Pure Chem. Sect.*, 1954, **75** [9], 962).—The dissociation constants of $Cu(CN)_4'''$, $Zn(CN)_4''$ and $Cd(CN)_4''$ were measured by the use of concentration cells. The *K* values found were $2·77 \times 10^{-28}$ (Cu), $1·75 \times 10^{-17}$ (Zn) and $9·09 \times 10^{-17}$ (Cd). K. SAITO

2044. **The use of ion exchange for the separation of copper, cadmium and zinc from thiosulphate solutions.** A. Vasil'ev, V. F. Toropova and A. A. Busygina (*Uch. Zap. Kazansk. Un-ta*, 1953, **113** [8], 91–102; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 44,488).—The ion-exchange separation of Cu, Cd and Zn is based on differences in the stabilities of the thiosulphate complexes of these metals. The concentrations of the solutions are determined polarographically. Under static conditions, adsorption of the metals on the Na form of Wofatit P decreases with increasing concn. of thiosulphate in the soln.; the effect decreases in the order Cu, Cd, Zn. Mixture of Cu and Zn (column washed with 0·1 *M* $Na_2S_2O_3$ at pH 7·6) and also Cu and Cd (column washed with 0·015 *M* $Na_2S_2O_3$ soln.) can be separated. E. HAYES

2045. **Polarographic determination of traces of copper, lead, zinc and iron in glass for pharmaceutical use.** A. Anastasi, E. Mecarelli and L. Novacic (*Ann. Chim., Roma*, 1955, **45** [1], 88–98).—The properties and behaviour of glass for pharmaceutical use are briefly discussed. A polarographic method for the determination of Cu, Pb, Zn and Fe in glass is described. This involves the elimination of silicates by heating with HF and $HClO_4$, dissolving the residue in HCl, and separation of Pb, Cu and Zn from Fe by means of dithizone. The individual metals are then determined by reduction at the dropping-mercury electrode. The analysis of a glass containing (0·5 to 1) $\times 10^{-5}$ parts of Cu, 1×10^{-5} parts of Pb, 5×10^{-5} parts of Zn, and $1·5 \times 10^{-3}$ parts of Fe is described. C. A. FINCH

2046. **Separation and determination of microgram quantities of silver, mercury and copper with dithizone.** H. Friedeberg (*Anal. Chem.*, 1955, **27** [2], 305–306).—By means of a fractional extraction technique, employing the partition of the dithizonates of Ag^{+}, Hg^{++} and Cu^{++} between CCl_4 and water at various pH values, these cations are quantitatively separated and then the complexes are photometrically determined. The soln. of mixed cations (pH 2 to 5) is extracted with successive portions of 13 p.p.m. of dithizone (in CCl_4) (**I**), and the Ag dithizonate is back-extracted into 10 per cent. NaCl - 0·015 *N* HCl (2 × 3-ml portions). This aq. phase is diluted to 60 ml with water and the Ag is extracted with **I** for photometric determination. The Hg^{++} and Cu^{++} remaining in the organic phase of the first partition are extracted into 6 *N* HCl (2 × 3-ml portions). The combined aq. extracts from this are adjusted to pH 1·5 to 2 (6 *N* aq. NH_3 and HCl) and are treated with 0·01 *N* EDTA (1 ml). Extraction of this soln. with **I** separates Hg^{++} into the org. phase, where it is determined photometrically. Cu^{++} is determined in the remaining aq. phase as follows: to it is added 0·1 *N* $CaCl_2$ soln. (1 ml), 25 per cent. ammoniacal ammonium citrate (at pH 9) (3 ml) and the mixture is adjusted to pH 9 with aq. NH_3. The Cu^{++} is extracted into **I** and the combined extracts are washed with water. Cu^{++} is back-extracted with 6 *N* HCl (3 ml) and the aq. phase is adjusted to pH 2 to 3 with aq. NH_3. Following a further extraction with **I** the Cu^{++} is determined photometrically. Slightly modified procedures are described where separation and determination of the metals may be achieved when one of them is in large excess, except in the case of preponderance of Hg^{++} over Cu^{++}. D. A. PANTONY

2047. **Quantitative analysis without separation. XI. Gravimetric analysis without separation for the systems Ag - Cu and Ag - Pb.** N. Unohara (*J. Chem. Soc. Japan, Pure Chem. Sect.*, 1954, **75** [9], 947–949).—The thermal decomposition curves of AgI, Cu_2I_2, PbI_2, AgI - Cu_2I_2 and AgI - PbI_2 were studied by means of a thermobalance. The quant. analysis of Cu and Pb in mixed halides with Ag from the weight difference (for Cu at 190° and 480° C, for Pb at 60° and 870° ± 5° C) can be used when the amounts of the ingredients are of comparable order.
K. SAITO

2048. **Chemical analysis of beryllium. II.** T. Akiyama (*Japan Analyst*, 1953, **2** [1], 13–17).—The following methods for the gravimetric analysis of Be are re-examined: (*i*) pptn. with NH_4Cl and aq. NH_3, (*ii*) pptn. of hydroxide through hydrolysis of a caustic alkali soln., (*iii*) pptn. with aq. NH_3 after the separation of Al with oxine and (*iv*) pptn. of hydroxide by hydrolysis of an acidic soln. of Be with a mixture of KI and KIO_3. The influence of experimental conditions is studied. K. SAITO

2049. **Determination of magnesium in cast iron.** I. Sajó and P. Répás (*Kohászati Lapok*, 1953, **8** [11], 225; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 40,009).—Magnesium is determined by titration with EDTA (disodium salt) with Eriochrome black T as indicator, after the iron has been pptd. electrolytically at a mercury cathode. The error is ± 0·005 per cent. E. HAYES

2050. **Polarographic determination of magnesium in coal ash and coke ash.** L. J. Edgcombe and G. R. E. C. Gregory (*Analyst*, 1955, **80**, 236–237).—The method of Stone *et al.* (*Ind. Eng. Chem., Anal. Ed.*, 1944, **16**, 596) for the determination of Mg has been applied to coal ash and coke ash. Solution of the ash and removal of group 3A metals are effected by the procedure of Edgcombe *et al.* (*Anal. Abstr.*, 1955, **2**, 873), the volume of the final soln. being 50 ml. To remove Mn and Pt, a 10-ml aliquot is evaporated to dryness with a small crystal of hydroxylamine hydrochloride in a silica crucible and heated at 450° to 500° C for 1 hr. The residue is dissolved by boiling with 5 ml of water. The soln. is mixed with 10 ml of an aq. NH_3 - NH_4Cl buffer soln. (0·036 *M* NH_4Cl adjusted to pH 10 with aq. NH_3) and 5 ml of standard oxine soln. (0·5 g. in 1 litre of 5 per cent. ethanol) and diluted with water to 25 ml. The product is set aside for 2 hr. with frequent shaking, then a suitable vol. in the polarographic cell is maintained at 25° C while N is passed in for 15 min. to remove O. The polarogram is made at sensitivity 1 in 15, damping position 3, from − 1·0 to − 1·6 V against a mercury-pool anode, and the wave height is recorded. A calibration graph is made in the same manner with a standard Mg soln. (MgO dissolved in the min. amount of HCl, diluted to $\simeq$ 100 μg per ml and standardised by pptn. and determination as $Mg_2P_2O_7$). The method is accurate for amounts of Mg up to 5 per cent. The presence of < 15 per cent. of CaO causes no interference. A. O. JONES

2051. **Amperometric titration. V. Determination of calcium and fluorine by an anodic ferrocyanide method.** O. A. Songina, A. P. Voĭloshnikova and M. T. Kozlovskiĭ (*Izv. Akad. Nauk Kazakh. SSR*, 1953, *Ser. Khim.*, [6], 69–77; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 45,094).—An anode ferrocyanide method for the amperometric determination of Ca^{++} and F' is based on the reaction between Ca^{++} and $[Fe(CN)_6]''''$. For the determination of F', an excess of Ca^{++} is added to the test solution and the excess is back-titrated in the presence of NH_4^+ without removal of the CaF_2 ppt. A rotating platinum micro-electrode is used. In the presence of NH_4^+, large amounts of Na^+ do not affect the determination, but Mg^{++} and Al^{+++} interfere. E. HAYES

2052. **An attempt to separate calcium isotopes by radiometric adsorption analysis.** R. Lindner (*Z. Naturforsch.*, 1954, **9a** [9], 798).—The separation of ^{45}Ca and ^{40}Ca is achieved on a hydrogen-loaded column of Dowex-50 resin (250 to 500 mesh) of 140-cm length and 11-mm diameter. The salt (as the carbonate) was dissolved in HCl. The column is heated to 95° C and the calcium is displaced with 5 per cent. aq. NH_3 (pH 8). Filtration is done under pressure to give a flow rate of 1 ml per sq. cm per min. Calcium appears in the eluate after 700 ml have passed through. The calcium band was collected fractionally in 40 ml. ^{45}Ca is more strongly adsorbed than ^{40}Ca and careful gravimetric and radiometric measurements show good separation, the activity of the recovered ^{45}Ca being higher than that of the original sample. E. KAWERAU

2053. **Method for the determination of calcium sulphate in mine water.** W. R. Harris (*S. Afr. Ind. Chem.*, 1955, **9** [1], 16–17).—A method of "reverse filtration" is employed to remove sludge from the sample. A measured vol. of the filtrate (5 ml) is added to industrial alcohol (15 ml) contained in a filter-paper resting in a funnel which is closed by a stopcock. After the $CaSO_4$ has pptd. in the filter-paper, the stopcock is opened and the ppt. is washed with more industrial alcohol. The filter-paper and ppt. are ignited in an electric muffle furnace, and the residue of $CaSO_4$ is weighed. A. M. SPRATT

2054. **Determination of microgram quantities of strontium in solution. Evaluation of flame-spectrophotometric method.** A. E. Taylor and H. H. Paige (*Anal. Chem.*, 1955, **27** [2], 282–284).—Full details of the flame-photometric method for the determination of strontium (0 to 10 p.p.m.) in water using the Beckman DU spectrophotometer with flame and photomultiplier attachments are described. The strontium soln. is atomised into an acetylene flame, and the flame intensity is measured at 681 mμ. Strontium is calculated from intensities of standards which give a linear concn. graph. In general, results are superior to those with a hydrogen flame and detection at 461 mμ. Fe, Na, Ca and Mg separately do not interfere. A precision of ± 0·5 p.p.m. is claimed. D. A. PANTONY

2055. **Polarographic determination of large amounts. Application to zinc ores.** W. Vinc[illegible]r (*Chim. Anal.*, 1955, **37** [4], 136–139).—From 40 to 70 per cent. of Zn in minerals, *e.g.*, graded zinc blende, can be determined polarographically to within 0·2 per cent. by making two measurements of current-intensity (*i*) at the two wave-limiting potentials (1·0 and 1·7 V) instead of recording the *i*-V curve. Devices for ensuring uniform discharge (60 drops per min.) from the capillary (30 mm long) and a const. temp. (25° ± 0·1° C) of the solutions are described. Damping of the galvanometer is effected with two electrolytic condensers, each of 4000 micro-farads and in series in opposition, arranged in parallel with the galvanometer. *Procedure*—To the soln. obtained by treating the sample (0·5 g) with HCl and H_2O_2, add 60 ml of aq. $(NH_4)_2CO_3$ and 20 ml of 0·4 per cent. aq. gelatin containing 0·1 per cent. of salicylic acid. Make the solution up to 500 ml and leave for 2 hr.; then transfer 10 ml of the clear soln. to the polarographic cell, adding sufficient mercury to provide an area of $\simeq$ 1 sq. cm. Leave the cell in the thermostat for 15 min. before taking the two galvanometer readings, first at 1·0 V and then at 1·7 V. Wait two min. before applying each potential to the cell and taking the reading. The zinc content is calculated by reference to the corresponding readings obtained with standard solutions of pure Zn. W. J. BAKER

2056. **Colorimetric determination of zinc by dithizone. The masking of interfering ions by 2-hydroxyethylammonium 2-hydroxyethyldithiocarbamate.** T. Kato and S. Takei (*Japan Analyst*, 1953, **2** [3], 208–210).—2-Hydroxyethylammonium 2-hydroxyethyldithiocarbamate (**I**) is synthesised from ethanolamine (6 g) and CS_2 (3·5 g) in methanol. The pH value of the zinc soln. ($\simeq$ 10 ml, 5 to 60 μg of Zn) containing other heavy metals and methanol soln. of **I** (7 per cent., 1·0 ml) is adjusted to 5·5 to 6·0 with acetate buffer. Zn is extracted with dithizone - CCl_4 (0·01 per cent.) and excess of dithizone is removed from the layer of CCl_4 with a soln. of Na_2S (0·05 per cent.) until the aqueous layer becomes colourless. The extinction of the CCl_4 layer is measured in the usual way. Thus the undesirable influence of Pb (< 0·5 mg), Cd (< 2 mg),

Bi (< 1 mg), Ag (< 0·5 mg) and Cu (< 0·3 mg) can be eliminated. Hg^{++} retards the extraction of Zn.
K. SAITO

2057. **Separation of small amounts of zinc by evaporation in a current of hydrogen.** W. Geilmann and R. Neeb (*Angew. Chem.*, 1955, **67** [1], 26–31).—The Zn is sublimed from the material by heating in a current of H at ≃ 1100° C and determined polarographically or colorimetrically with dithizone. The sample (0·1 to 10 g according to Zn content) is placed in an unglazed porcelain boat, which is placed in a quartz sublimation-tube heated electrically to ≃ 1100° C. A current of H is passed through for 2 hr. The Zn, which condenses in the cool capillary part of the tube, is dissolved out with a few drops of hot aq. HCl (1 : 1) followed by 30 per cent. H_2O_2 and water, and the Zn content of the solution is determined. If Pb is present, the sublimate is dissolved in HNO_3 and the solution evaporated to dryness with HCl. The process is applicable to bauxite, alumina, Al metal, soils, etc.
S.C.I. ABSTR.

2058. **New gravimetric method for the determination of mercury as metal.** S. Cosma (*Studii si Cercetări Chim.*, 1953, **1**, 73–79; *Referativnŷĭ Zh., Khim.*, 1954, Abstr. No. 40,016).—Hexamine (1 g) is dissolved in a hot soln. (25 to 45 ml) of mercury salt containing 2 to 3 ml of conc. HCl; 20 to 40 ml of a 20 per cent. soln. of NaOH are then added with vigorous stirring until the soln. becomes cloudy. When a grey ppt. of metallic Hg has formed, the cooled liquid is filtered and the ppt. is washed, first with water to remove Cl′, and then with ethanol and ether; it is finally dried in a vacuum-desiccator. Insoluble Hg salts are dissolved in HCl, hot aqua regia or hot NaCl soln. The method is as precise as that in which Hg is determined as HgS and it is applicable to complex mercury compounds.
E. HAYES

2059. **Estimation of metallic mercury on the surface of tinned copper.** G. T. Kerr, S. S. Macut and C. C. Neely (*Anal. Chem.*, 1955, **27** [2], 294–295).—Samples (≃ 0·7 g) of tinned copper are coated with a known wt. of Hg (1 to 3 mg) by a standard procedure. The Hg is removed from the weighed coated samples at 190° C under 1 mm pressure. The Hg deposit is obtained by the loss in wt. An error of ± 0·21 mg is given.
D. A. PANTONY

2060. **Determination of radium-B in radioactive mineral water.** S. Umemoto (*Japan Analyst*, 1953, **2** [3], 201–205).—A method of determining micro amounts of Ra-B (isotope of Pb) in mineral water of weakly radioactive mineral springs was studied with special attention to the separation with Ra-A (isotope of Po) and Ra-C (Bi), and to the removal of traces of radon. The interference of H_2S is eliminated by the addition of aq. Br soln. Ra-A is removed by shaking the sample soln. with dithizone - CCl_4 soln. in an acidic medium (pH ≃ 1). Ra-B is extracted by the same reagent from a basic soln. (pH 9 to 10) in the presence of NaCN. The CCl_4 layer is dried quickly on a piece of filter-paper and the β-activity measured with a Lauritsen electroscope. This method enables the determination of as little as 20×10^{-10} curie of Ra-B per litre.
K. SAITO

2061. **Spectrochemical evaluation of boron in minimal concentrations.** M. V. de la Pina and A. Camunas (*An. Real Soc. Esp. Fis. Quim.*, 1955, **51B** [2], 169–172).—The variations of the method of Rivas (*Angew. Chem.*, 1937, **50**, 903–911), including the "absolute method" of van Calker (*Spectrochim. Acta*, 1944, **2**, 233), are discussed, with the errors for solutions of different concentration. When the absolute method is used, the NaCl, KNO_3, K_2SO_4, $Ba(NO_3)_2$, HCl, H_2SO_4 and aq. NH_3 have no effect up to high concn. Curves are given for the determination of amounts of 0·003 to 0·068 μg of B by using lines at 2496·78 A and 2497·73 A generated at a silver electrode by a high-tension condensed spark, and the determination of 0·47 to 5·08 μg of B by using the same lines generated at a graphite electrode by an arc.
D. LEIGHTON

2062. **Studies on the chemical analysis of boron. III. Paper chromatography of boron.** S. Muto (*J. Chem. Soc. Japan, Pure Chem. Sect.*, 1954, **75** [9], 949–951).—The paper-chromatographic distribution of BO_3''' was studied with various solvents. The R_F values are ≃ 0 in solvents that are immiscible with water and 0·6 to 1·0 in those that are miscible. The stronger the acid that is added to the org. solvent (*e.g.*, ethanol), the greater is the R_F value. In general, the addition of bases does not affect the R_F value, but aq. NH_3 causes a significant decrease. Addition of mannitol gives no change in R_F. The influence of other salts was also studied.
K. SAITO

2063. **Determination of boric acid in glass and enamel.** E. Eipeltauer and G. Jangg (*Öst. Chem-Ztg*, 1955, **56** [4], 97–99).—After a review of the usual volumetric determinations of boric acid contents in technical glass and enamels, complicated by the interference of admixtures (Al, Fe, Ti, Sn, Pb, SiO_2, etc.), a new method is described, by means of which interfering admixtures are pptd. with NaOH in the presence of excess of $BaCl_2$ at pH 8; the filtrate containing borates is titrated acidimetrically with an error of ± 1 per cent. As an example, powdered glass (0·2 to 0·5 g), fused with Na_2CO_3 and lixiviated with H_2O, is slightly acidified with HCl and carefully heated to expel CO_2. After the addition of 10 ml of 20 per cent. $BaCl_2$ solution and a few drops of phenolphthalein, the heated solution is stirred and treated dropwise with ≃ 2 *N* NaOH to the change of indicator. The ppt. is filtered off warm, dissolved in little warm HCl (1 : 3) and the pptn. is repeated as before. The volume of the filtrates containing the borates should not be greater than 150 ml. S.C.I. ABSTR.

2064. **Micro-quantitative determination of carbon, particularly in tantalum carbide.** V. I. Smirnova and B. F. Ormont (*Zh. Anal. Khim., SSSR*, 1954, **9** [6], 359–363).—Carbon dioxide from the combustion of the carbon-containing material is absorbed in a weighed amount of barium hydroxide solution in a special absorption tube (*Anal. Abstr.*, 1955, **2**, 2253) and the excess of alkali is titrated with 0·1 *N* HCl, using a weighing burette.
G. S. SMITH

2065. **Polarographic determination of small quantities of carbon monoxide.** J. Vykoukal and K. Linhart (*Paliva*, 1953, **33** [11], 236–241; *Referativnŷĭ Zh., Khim.*, 1954, Abstr. No. 41,710).—Methods of determining CO in mine gases are critically reviewed. In a proposed polarographic method, CO is oxidised by I_2O_5 at 110° C, the liberated iodine is absorbed by NaOH soln. and the iodide formed is oxidised by ozone to iodate; the iodate is then determined polarographically. The method permits the determination of 0·00006 per cent. of CO in a 200-ml sample of gas; the error is ± 0·5 to 10 per cent., according to the concn. of CO.
E. HAYES

2066. **Indirect colorimetric determination of titanium.** T. Nozaki (*J. Chem. Soc. Japan, Pure Chem. Sect.*, 1954, **75** [9], 937–939).—Titanium (0·01 to 1 mg) can be pptd., on a micro scale, in a 0·5 *N* H_2SO_4 soln. with microcosmic salt. The ppt. is washed with 5 per cent. NH_4NO_3 soln. and dissolved in a small amount of *N* KOH and saturated Na K tartrate soln. Phosphorus is determined colorimetrically by the usual method and the amount of Ti is calculated. The standard deviation from the mean is $\simeq \pm 1{\cdot}46$ per cent. for 0·2 to 1·0 mg of Ti and $\simeq \pm 2{\cdot}28$ per cent. for 0·01 to 0·1 mg of Ti.
K. SAITO

2067. **Determination of hydrogen in titanium.** T. D. McKinley (*J. Electrochem. Soc.*, 1955, **102** [3], 117–123).—A description is given of the construction, calibration and operation of an apparatus for the simple rapid determination of 0·001 to 0·33 per cent. of H in Ti with a probable error of $\simeq 5$ per cent. The method is based on the measurement of the equilibrium pressure of H over the metal in a closed system under predetermined conditions. The physical form of the specimens is unimportant.
A. JOBLING

2068. **The photometric determination of zirconium in Hiduminium RR.350 and similar alloys.** E. C. Mills and S. E. Hermon (*Metallurgia*, 1955, **51** [305], 157–158).—A photometric method has been devised for the routine determination of Zr in Al alloys. *Procedure*—A 1·5-g sample is weighed into a conical beaker, covered with 15 ml of water, and 15 ml of conc. HCl (11·3 *N*) are added. The beaker is then covered with a watch-glass and allowed to stand in cold water until the violent reaction has ceased. A further 30 ml of acid is added, the beaker is washed down inside with water, and transferred to a hot-plate. It is then heated for 15 min., during which the sample should be boiling for about 10 min. After cooling, the sample is transferred to a 100-ml calibrated flask, the solution is diluted to the mark and mixed. The slight Cu ppt. is allowed to settle, or, if a noticeable cloudiness persists after 5 to 10 min., approx. 30 ml of the solution are filtered off through a small dry Whatman No. 541 filter-paper into a dry beaker. With a pipette, 10 ml of the sample are measured into the original beaker and a second 10-ml aliquot into a centrifuge tube, tall beaker or boiling-tube. To the 10-ml aliquot in the original beaker bromophenol blue indicator is added; after being slightly diluted, the solution is titrated with 1·13 *N* Na_2CO_3 solution to the blue end-point. This titration value divided by 10 gives the ml of 11·3 *N* hydrochloric acid in the aliquot, and 3·00 minus this figure gives the addition of acid required. This volume of acid is added to the second aliquot from a burette, 10 ml of alizarin S soln. (0·15 per cent. w/v) are added, and the solution is diluted to 25 ml with water. After a gentle shaking, the tube is transferred to a boiling-water bath for 5 min., cooled rapidly, transferred to a 100-ml calibrated flask containing 10 ml of 20 per cent. v/v hydrochloric acid, then diluted to the mark and mixed. The extinction value of the solution is measured with a Spekker absorptiometer (H760) using Ilford yellow-green No. 605 filters and a zero setting of 0·80 on a processed Zr-free alloy sample. This method is suitable for foundry control purposes and, with modification, for the determination of Zr in pure Al.
G. C. JONES

2069. **Detection of a micro-amount of germanium by the use of an ion exchanger. I.** H. Kakihana and T. Murase (*J. Chem. Soc. Japan, Pure Chem. Sect.*, 1954, **75** [9], 907–914).—The sensitivity of the spot test for Ge with haematoxylin is enhanced by the use of a piece of Amberlite IRA-411 (Cl or F form). A distinctive purple colour is revealed on the surface of the resin by 0·010 μg of Ge in 0·02 to 1 *N* HCl (limit of concn. 0·35 p.p.m.). The interference of Sb^{III}, Fe and Bi can be eliminated by putting a piece of Amberlite IR-120 (Na form) into the sample soln. With the exception of Sn, no other elements interfere.
K. SAITO

2070. **Use of alternating-current arcs in the spectroscopic quantitative analysis of bronze.** R. Baistrocchi and L. Gazzi (*Chim. e Ind.*, 1955, **37** [3], 175–176).—The use of an a.c. arc as an exciting source for the determination of Sn in artistic and archaeological bronzes by emission spectrography is described. The technique is applicable to a wide range of concn., with an error of < 6 per cent.
C. A. FINCH

2071. **Determination of lead in lead drosses and lead-base alloys. Applications of ethylenediaminetetra-acetic acid method.** J. L. Pinkston and C. T. Kenner (*Anal. Chem.*, 1955, **27** [3], 446–447).—The sample containing 0·4 to 0·48 g of Pb is dissolved in H_2SO_4 (20 ml) and org. matter is destroyed with HNO_3. The soln. is cooled and diluted to 200 ml, and digested with tartaric acid (2 g). After being cooled, filtered off and washed, the ppt. is dissolved in 45·4 per cent. aq. ammonium acetate (30 ml) and diluted with water (200 ml). To this soln. is added tartaric acid (2 g) and conc. aq. NH_3 (25 ml). The soln. is titrated, at 70° to 80°C, with 1·86 per cent. EDTA (disodium salt), with Eriochrome black T as indicator. Results are presented for analyses of 7 lead-bearing materials. A mean standard deviation of $\pm$ 0·11 per cent. is claimed. Ca^{++} and Ba^{++} interfere.
D. A. PANTONY

2072. **Gravimetric determination of lead with disubstituted dithiocarbamates.** E. Bremanis, L. Schaible and K. G. Bergner (*Z. anal. Chem.*, 1955, **145** [1], 18–23).—Lead is determined gravimetrically in the presence of other heavy metals by pptn. with dithiocarbamates, *e.g.*, the sodium salt. The method is generally more rapid than the classical sulphate pptn., and is very accurate for amounts of Pb > 2 mg. The heavy metals As, Sb, Sn, Cu, Co, Ni, Fe and Zn do not interfere. When Cd, Ag and Bi are present, treatment with HNO_3, dissolution and repptn. are necessary. Tl is quant. pptd. with Pb; Hg and Mg interfere; the behaviour of Au and Pt was not investigated.
D. R. GLASSON

2073. **Determination of lead in the tin of food-container tinplate.** A. Zvoníčková (*Průmysl Potravin*, 1953, **4** [11], 490–491; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 41,701).—The tin is removed from a weighed sample of tinplate by means of a 1 per cent. soln. of Sb_2O_3 in conc. HCl; the tinplate is washed, dried and re-weighed and the wt. of tin is found from the difference. The soln. is diluted with an equal vol. of water, filtered and the filtrate is evaporated to dryness; the residue is evaporated to dryness with 2 ml of conc. HNO_3 and 5 ml of conc. HCl, and this process is repeated 3 to 4 times. A few drops of conc. HCl, 3 ml of 40 per cent. acetic acid soln., 7 ml of a 25 per cent. soln. of ammonium acetate and a few drops of methyl red soln. are added to the final dry residue. The soln. is made up to 25 ml and the Pb in a 10-ml aliquot of this soln. is determined polarographically at 0·2 V.
E. HAYES

2074. **Estimation of thorium by organic reagents. V. Separation of thorium from uranium and their co-determination by 2 : 4-D.** S. K. Datta and G. Banerjee (*J. Indian Chem. Soc.*, 1954, **31** [12], 929–932).—Complete separation of Th from U can be effected by 2:4-D (2:4-dichlorophenoxyacetic acid) at pH 2·6 to pH 3·4, by single precipitation, when the ratio Th:U is 1:1. When the proportion of U is higher, double precipitation is essential and good results are given in the same pH range up to a Th:U ratio of 1:26. Uranium can be quant. recovered from the filtrate by the sodium salt of 2:4-D at pH $> 5·0$. The use of this reagent in the co-determination of Th and U at definite pH ranges is suggested. Free 2:4-D gives no precipitate with U. Experimental details are given.

C. H. WHITTON

2075. **Polarographic determination of nitrates and nitrites in salt and meat-pickling salt.** R. Pletikha and E. Krzhizhova (*Zh. Anal. Khim., SSSR*, 1954, **9** [6], 366–372).—Nitrates and nitrites in pickling salts not containing sugars and proteins are determined by Kolthoff's polarographic method, but with uranyl acetate instead of the chloride. In the presence of sugars, nitrites are determined in acetic acid solution, without addition of uranyl acetate, from the height of the wave corresponding to the reduction of oxides of N, which begins at $-0·6$ V with reference to the saturated calomel electrode. In the presence of proteins a preliminary separation with alcohol is necessary before a nitrate determination by the uranyl method. The use of alcohol is unnecessary in the determination of nitrites by the acetic acid method.

G. S. SMITH

2076. **Volumetric determination of phosphorus.** D. M. Zall, E. Wagman and N. Ingber (*Anal. Chem.*, 1955, **27** [2], 277–279).—A soln. containing $\gtrsim 0·15$ g of Na_2HPO_4 and $\gtrsim 0·15$ g of Na_2CO_3 is prepared as follows: (*a*) *Boiler compound*, a suitable wt. is dissolved in water (100 ml). (*b*) *Cast iron*, a sample (5 g) is dissolved in dil. HNO_3 (1 + 1) (50 ml) and a few drops of HF; the soln. is taken to fumes with conc. $HClO_4$ (35 ml). After most of the $HClO_4$ has been volatilised, the residue is dissolved in hot water (75 ml), the soln. is filtered and run slowly into hot 50 per cent. NaOH (150 ml). The suspension is cooled and made up to 500 ml. A portion of soln. is decanted and filtered, and 200 ml of it are neutralised with dil. H_2SO_4 (1 + 3) (phenolphthalein), an excess (5 ml) of acid then being added. The soln. is boiled, cooled and carefully neutralised with NaOH (phenolphthalein). Alternatively, Fe may be removed by electrolysis at the mercury cathode in $HClO_4$ medium. (*c*) *Organo-phosphorus compounds*, the sample (0·2 to 1 g) is ignited in O in a bomb, and the residue is dissolved in water. After being made alkaline with NaOH, the soln. is filtered as under (*b*). In each case the soln. is either titrated directly with 0·05 *N* H_2SO_4 (methyl purple), or a known (excess) vol. of 0·05 or 0·1 *N* H_2SO_4 is added and the excess is back-titrated with 0·05 or 0·1 *N* NaOH (methyl purple). In all titrations, entry of CO_2 must be avoided, either by boiling or by flushing with CO_2-free N. The P content is calculated from a given formula. Results are presented for 12 synthetic samples, 10 boiler-compound samples and 3 standard cast irons. They compare favourably with those from the standard gravimetric procedure.

D. A. PANTONY

2077. **Direct-recording spectrographic determination of phosphorus in steel and iron ore.** C. G. Carlsson and L. Danielsson (*Jernkontor. Ann.*, 1954, **138** [7], 383–403).—The method is based on the use of a spectrograph with narrow dispersion (Hilger medium quartz spectrograph) in order to obtain a sufficient light strength. A slit 12 μ × 2 mm is used, with a photocell 1 P 28 (R. C. A.). Owing to the close proximity of the Cu and P lines very accurate setting and focusing is necessary. There is some overlap of the peak absorptions for Cu (2148·97 A) and P (2149·11 A), and the slit must be adjusted to one side of the P maximum. The exact position is determined by calibration with 2 samples of low copper content, one high and one low in P. The sensitivity and zero of the recording equipment are then adjusted so that the phosphorus content is read direct on the scale. A sample with known phosphorus content and maximum copper content (*e.g.*, 0·4 per cent.) is tried. If the result is too high the slit is moved away from the Cu line by a few μ. This is repeated until Cu does not interfere. For a routine check, the slit is first adjusted to the centre of the Cu line, then moved according to the scale to the position as found previously. For a reference line for standardisation of the photocell the Fe line is used (2253·1 A). The electrodes are two rods of the sample, or a flat surface of it, and a triggered low-tension spark was found satisfactory. The P line at 2136·20 A, which is farther removed from a Cu line, may also be used, so that it is possible to adjust on the centre of the P line and the slit may be widened, but an objection is the lower intensity of this line which makes it necessary to use maximum amplification for phosphorus contents $< 0·01$ per cent. The reproducibility for P in steel (0·005 to 0·1 per cent.) is good, with a standard deviation of ± 5 per cent. The time required is 2 minutes.

G. MIDDLETON

2078. **Colorimetric determination of phosphorus by modified molybdophosphate method.** D. N. Bernhart and A. R. Wreath (*Anal. Chem.*, 1955, **27** [3], 440–441).—One g of organic or polyphosphate containing small proportions of PO_4''' is dissolved in acetone or water (100 ml), respectively. To an aliquot (1 to 5 ml) are added 3·76 per cent. ammonium molybdate in dil. (3 + 7) H_2SO_4 (2 ml), acetone (10 ml) and either acetone or water to 25 ml. The absorption is measured at 430 mμ *vs.* a blank and compared with standards. Procedures are given for the determination of total P_2O_5 in inorganic or organic phosphates. Results are presented for 17 phosphate analyses.

D. A. PANTONY

2079. **Use of nuclear emulsions to determine small amounts of ^{33}P present in samples of ^{32}P.** J. Mayr (*Experientia*, 1955, **11** [1], 21–22).—The method is based on counting the numbers of densely ionised terminal parts of the β-ray tracks in nuclear emulsions. The emulsion thickness of 200 μ was chosen to include almost the whole length of the tracks from ^{33}P while recording only a small proportion of the tracks of ^{32}P. Direct measurements of the radio-activities and absorption curves in aluminium are made.

R. S. TONKS

2080. **A note on the direct estimation of arsenous and antimonous oxides by standard potassium dichromate.** S. Gireesan and A. Visvanathan (*J. Indian Chem. Soc.*, 1954, **31** [8], 643–644).—In the determination of As and Sb by means of standard potassium dichromate, the addition of sodium bicarbonate to the acidified mixture of potassium

dichromate and iodine causes pptn. of colloidal chromium hydroxide, which adsorbs some of the free iodine. This adsorption can be almost completely avoided by first removing most, but not all, of the iodine from the acid medium with the titrating solution, and then reducing its concentration by dilution before adding the bicarbonate. The presence of Rochelle salt sequesters most of the Cr^{***} in a soluble complex and gives an easily visible end-point. Results differ by less than 1 per cent. from the correct value. R. J. MAGEE

2081. **Radioactivation analysis of arsenic in silicon.** J. A. James and D. H. Richards (*Nature*, 1955, **175** [4461], 769–770).—Radioactivation analysis overcomes dependence on purity of reagents for the determination of traces of Group V elements in semiconductors. Provided that the impurities yield radioactive isotopes these atoms may then be separated with added carrier and laboratory-grade reagents. Thermal neutron irradiation has been applied to the determination of As in Si. The reactions $^{30}Si\ (n,\gamma)\ ^{31}Si \xrightarrow{\beta^-} {}^{31}P$ and $^{75}As\ (n,\gamma)\ ^{76}As \xrightarrow{\beta^-\gamma} {}^{76}Se$ take place. After the Si is dissolved in NaOH soln. in the presence of H_2O_2, with As_2O_3 as carrier, the solution is reduced in bulk and Smales and Pate's method (*Brit. Abstr. C*, 1952, 378) is used for the separation of As. The chemical recovery of the arsenic carrier is 80 to 90 per cent. At a pile factor of 10, an arsenic content of 2 to 3 p.p.m. gives 9000 counts per min. from a 0·2-g sample on the third day on shelf 2 of a lead castle. The detection limit is 0·0003 p.p.m. on a 1-g sample. In practice, arsenic contents of 0·0001 to 3 p.p.m. have been detected. G. A. BASSETT

2082. **Modified iodimetric estimation of vanadium.** M. R. Verma and V. M. Bhuchar (*J. Sci. Ind. Res. India, B*, 1955, **14** [1], 19–24).—Quinquevalent V is reduced with potassium citrate in acidic medium to the quadrivalent state, treated with an excess of iodine and back-titrated with sodium arsenite. The method is suitable for the determination of 4 to 40 mg of V_2O_5 and is more satisfactory in certain cases than the SO_2 reduction method or the ferrous ammonium sulphate method. R. J. COLE

2083. **Simultaneous absorptiometric determination of tantalum and niobium in ores.** A. E. O. Marzys (*Analyst*, 1955, **80**, 194–203).—The thiocyanate - acetone method for the determination of Nb is combined with a modified procedure for determining Ta with pyrogallol so that both determinations are made with one sample soln. The titanium content of another aliquot of the soln. is determined for use in applying a correction. Solution of the ore is effected by fusion with $NaHSO_4$ (preceded by treatment with HF for highly silicious ores), and aq. tartaric acid is used as the extractant in the pyrogallol method for determining Ta. Addition of certain amounts of HCl and ammonium oxalate to the tartaric acid extract eliminates niobium interference, reduces titanium interference and gives a max. absorption peak for Ta at 350 mμ. The procedures for determining Nb and Ti are slightly modified forms of those described previously (*Anal. Abstr.*, 1954, **1**, 2364). There is no interference from other elements commonly occurring in these ores.
A. O. JONES

2084. **The determination of oxygen in beryllium by the vacuum fusion method on a micro scale with a note on the determination of oxygen in zirconium.** J. N. Gregory and D. Mapper (*Analyst*, 1955, **80**, 230–236).—Oxygen present as an impurity in beryllium can be determined by means of the micro-scale vacuum fusion apparatus (Gregory *et al.*, *Anal. Abstr.*, 1955, **2**, 2282) in samples of 2 to 10 mg containing as little as 0·2 per cent. of O. Rigorous control of the experimental conditions is necessary with exact temp. control and use of a molten-platinum bath in the graphite crucible. Hydrogen, evolved at a very much lower temp., was found in all samples of Be analysed, and can also be determined by the method described. Attempts to determine O and N in Zr with the apparatus previously described (*loc. cit.*) were unsuccessful; with the modified apparatus O was readily determined in Zr. A. O. JONES

2085. **Quantitative analysis of mixtures of hydrogen sulphide and sulphur dioxide.** B. Smith (*Chalmers Tek. Högsk. Handl.*, 1955, [150], 19 pp.)—The classical Lunge and Winkler methods for analysis of mixtures of H_2S and SO_2 are critically discussed. The former method is not satisfactory as it gives low results for both gases. A more accurate Winkler method is described in which H_2O_2 is used as the absorbent for SO_2 and larger amounts of gas are used for analysis. It is also found that the presence of pumice in the $CuSO_4$ absorbent for H_2S is not essential. The $CuSO_4$ absorbent is dried for 10 hr. at 140° C and placed in a U-tube which is heated in a paraffin bath at 90° to 100° C. The gas is passed through, the U-tube is wiped, cooled in a vacuum-desiccator, then thoroughly cleaned and weighed. The gain in wt. is a measure of the H_2S content of the gas. The gas is then passed through a Dehydrite tube and then into a Grote - Krekeler vessel containing 6 per cent H_2O_2. The contents of the vessel are titrated with 0·05 *N* NaOH (methyl red) to give a measure of the SO_2 content of the gas.
S.C.I. ABSTR.

2086. **Sulphate determination with barium chromate.** O. Hauser (*Mitt. Chem. Forsch. Inst. Wirts. Öst.*, 1955, **9** [1], 1–2).—A method of determining sulphate by pptn. with a weakly acid soln. of $BaCrO_4$ is described; the errors likely to arise in the gravimetric determination with $BaCl_2$ are thus avoided. The neutral sulphate soln. is treated at the b.p. with an excess of $BaCrO_4$ slightly acidified with HCl and boiled for 1 min. After cooling somewhat, aq. NH_3 is added in slight excess, which is removed by boiling. The ppt. of $BaSO_4$ + $BaCrO_4$ is filtered off and washed with a little water. The cooled filtrate is treated with KI soln. and conc. HCl (5 ml), and the liberated iodine is titrated with $Na_2S_2O_3$. 1 ml of 0·1 *N* $Na_2S_2O_3$ ≡ 3·2695 mg of H_2SO_4. R. J. MAGEE

2087. **Conductometric titration of sulphuric and hydrochloric acids and their mixtures in anhydrous acetic acid.** T. Higuchi and C. R. Rehm (*Anal. Chem.*, 1955, **27** [3], 408–411).—Mixtures of H_2SO_4 and HCl in anhyd. acetic acid are made up to 100 ml with that solvent in a conductivity cell at constant temp. The titration with standard acetate, especially of the Li salt, is performed conductometrically. The curve for specific conductivity *vs.* ml of titrant shows three distinct changes of direction, corresponding to the equivalents of Cl', SO_4'' and HSO_4'. The relative merits of Li, Na, K and tripentylammonium acetates as titrating agents are discussed. D. A. PANTONY

2088. **Spectrophotometric determination of chromium (III) with the disodium salt of ethylenediaminetetra-acetic acid.** R. Fernández Cellini and E. Alonso Valiente (*An. Real Soc. Esp. Fís. Quím.*, 1955, **51 B** [1], 47–52).—Tervalent Cr cations form a colour with EDTA which is violet in acid solution, changing to blue in alkaline solution. The colour has highest absorption at 538 mμ and pH 2 to 4. A solution containing up to 100 μg of Cr per 15 ml is adjusted to pH 2 to 4, 2 ml of a solution of EDTA (13·3 g per litre) are added, the mixture is boiled for 10 min., cooled, and made up to 15 ml. The absorption is measured in a 5-cm cell at 538 mμ, with water as a blank. The concn. of Cr is read from a calibration graph. D. LEIGHTON

2089. **Semi-micro determination of chromium in chrome leather.** N. W. von Bassenheim (*Industr. y Quím.*, 1954, **16** [8], 468–469).—About 10 mg of finely divided leather are accurately weighed into a 50-ml Erlenmeyer flask and moistened with 2 ml of water; 3 ml of $HClO_4$ are added, and oxidation is effected by gentle heating, losses being prevented by placing a funnel in the flask-mouth. Oxidation is assumed to be complete when the colour of the mixture is orange - red, owing to the formation of $Na_2Cr_2O_7$. The funnel is washed with water, the liquid is cooled and made up to 15 ml with water, then boiled gently till no more Cl can be detected in the vapours evolved (starch - iodine indicator paper). After cooling, addition of 1 ml of HCl, re-cooling and addition of 5 ml of 10 per cent. KI soln., the flask contents are titrated with 0·1 *N* $Na_2S_2O_3$ from a microburette, with soluble starch as indicator. The Cr_2O_3 is calculated from the volume required. Comparison of results with those obtained by the A.L.C.A. macro-method shows a maximum difference for Cr_2O_3 of 0·11 per cent.; 19 out of 20 results were within 0·08 per cent., 12 within 0·05 per cent., and 6 within 0·02 per cent. Al_2O_3 in the leather can also be determined by pptn. from the Cl-free solution obtained as above, either with aq. NH_3 soln. or as the 8-hydroxyquinoline complex, Cr being determined in the filtrate plus washings. D. LEIGHTON

2090. **A new spectrophotometric determination of chromium and cobalt with [their double complex of] ethylenediaminetetra-acetic acid.** H. Goto and J. Kobayashi (*J. Chem. Soc. Japan, Pure Chem. Sect.*, 1954, **75** [9], 964–968).—A complex salt which is formed from Co^{++}, Cr_2O_7'' and EDTA (**I**) has two absorption max., at 558 and 380 mμ, which can be used for the colorimetric determination of both Co^{++} and Cr_2O_7'' in 0·5 *N* acetic acid. The extinction coeff. - concn. plots are straight lines for 0 to 40 μg of Cr per ml and for 0 to 100 μg of Co per ml. Fe^{+++}, Ni^{++} and Cu^{++} interfere because of similar absorptions of their **I** complexes. The interference of Al^{+++}, Zn^{++}, Cd^{++}, Ba^{++} and Pb^{++} can be eliminated by adding an excess of the disodium salt of **I**. K. SAITO

2091. **Spectrophotometric determination of molybdenum and its application in the analysis of molybdophosphates.** B. Ricca and G. D'Amore (*Ann. Chim., Roma*, 1955, **45** [1], 69–80).—The spectrophotometric behaviour of molybdoferrocyanides, of general formula $M_4Fe(CN)_6.2MoO_3$, is studied, to provide a method for determining small quantities of Mo. The effect of concn. and time on the development of colours between 450 and 700 mμ is examined. Ions containing P, As, Si, Cr or W interfere with the reaction, but not those containing Cl, S, N or Al. When this spectrophotometric determination is applied to the analysis of samples of ammonium molybdophosphate, the classic method of Woy is shown to give a product purer than that from Frey's method (*Brit. Abstr. C*, 1950, 479). C. A. FINCH

2092. **Separation of uranium from tervalent iron by ion exchange.** R. Klement (*Z. anal. Chem.*, 1955, **145** [1], 9–12).—Uranyl ions are separable from Fe^{+++} by chromatographing on the cation-exchange resin Lewatit S 100 using 0·8 *N* HCl as the eluent. Amounts of $Fe^{+++} < 200$ mg are separated from mixtures containing U : Fe ratios of 1 : 0·6 to 1·6 by columns < 55 cm in length and 10 mm in diameter. The U is determined to an accuracy of ± 1·5 per cent. Any Cu present is separated from U by the use of 0·6 *N* HCl as eluent. D. R. GLASSON

2093. **A note on chloramine B as a volumetric reagent. Volumetric estimation of difficultly soluble iodides.** A. Singh (*J. Indian Chem. Soc.*, 1954, **31** [8], 648).—The use of chloramine B as a standard volumetric reagent for the determination of lead iodide, silver iodide and mercurous iodide is outlined. R. J. MAGEE

2094. **Colorimetric determination of manganese. Oxidation with bromate in sulphuric acid medium.** W. C. Purdy and D. N. Hume (*Anal. Chem.*, 1955, **27** [2], 256–258).—Manganese is oxidised in 8 *N* H_2SO_4 soln. with BrO_3' to Mn^{+++}, of which the colour intensity is measured at 500 mμ spectrophotometrically. The sample, containing 2 to 70 mg of Mn^{++}, is dissolved in < 50 ml of dil. H_2SO_4. To the soln. is added sufficient H_2SO_4 to make the final soln. 8 *N* with respect to H_2SO_4, 0·5 *M* KCN (2 ml) and 0·17 *M* $KBrO_3$ (5 ml); after being shaken, the mixture is made up to 100 ml with 8 *N* H_2SO_4 and, following the absorption measurement, Mn^{++} concn. is calculated from standards. Beer's law holds, and the colour is stable for one week. Reducing agents and Cr^{+++} interfere, but difficulties due to ions with insoluble sulphates can be avoided by filtration. Co^{++}, Ni^{++}, Zn^{++}, Fe^{+++}, Al^{+++}, Cu^{++}, Sn^{++++}, As^{+++}, Cd^{++}, Bi^{+++}, Ce^{+++}, Ce^{++++}, NO_3', F', Br' and Cr_2O_7'' do not interfere when present in the same concn. as the Mn^{++}. D. A. PANTONY

2095. **Determination of permanganate in the presence of dichromate by thallous sulphate.** U. Veereswara Rao, U. Muralikrishna and G. Gopala Rao (*Z. anal. Chem.*, 1955, **145** [1], 12–16).—The permanganate - dichromate mixed solution is run into standardised Tl_2SO_4 containing 1·2 *N* HCl and 1·2 *N* NaF. The visual end-point colour change from deep yellow to reddish-brown affords accurate results for solutions with $K_2Cr_2O_7 : KMnO_4$ ratios of < 2 : 1; larger amounts of $K_2Cr_2O_7$ mask the colour of the excess of unchanged $KMnO_4$. Electrometric methods permit analysis of 0·1 *N* $KMnO_4$ and $K_2Cr_2O_7$ solutions within ratios of 1 : 19 to 19 : 1. D. R. GLASSON

2096. **Colorimetric analysis without previous extraction. III. Determination of iron with cupferron.** F. Buscaróns and J. L. Marín Malumbres (*An. Real Soc. Esp. Fís. Quím.*, 1955, **51 B** [2], 117–120).—The determination of Fe with cupferron is studied, with methanol or butane-2 : 3-diol added to keep the complex in solution. A procedure is evolved to produce a solution whose colour is stable for several days and to boiling, and having a sensitivity of 5 μg, the dilution limit being 1 in 200,000. *Procedure*—To the neutral or slightly acid test solution is added 8 to 10 per cent. by vol. of conc.

HCl, and double the original vol. of methanol or an equal vol. of butane-2:3-diol. The mixture is stirred and 6 per cent. aq. cupferron is added until there is no intensification of colour. Alternatively, the amount of reagent to be added may be found experimentally by omitting the organic solvent and adding until white needles appear. This amount, plus 20 per cent. excess, should be used in the colorimetric procedure. D. LEIGHTON

2097. **Polarographic determination of iron in water-glass.** I. A. Korshunov and O. P. Malkova (*Uch. Zap. Gor'kovskogo Un-ta*, 1953, No. 24, 21–24; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 43,490).—A weighed sample of water-glass (2 to 3 g) is dissolved in 30 ml of conc. HCl, the soln. is evaporated to dryness, and the residue is extracted with 1·5 to 2 ml of conc. HCl and 10 ml of boiling water. The ppt. is filtered off from the cooled soln. and washed with boiling water. Conc. HNO_3 (2 to 10 drops) is added to the soln. and it is then made up to 10 to 100 ml (depending on the iron content) in a calibrated flask. Hydrogen is passed through 5 to 10 ml of the soln. in the electrolyser for 20 min. and it is then polarographed at 0 V. The iron content should be less than 5×10^{-3} mol. per litre and the HCl concn. should be between 0·2 and 2 *N*. Copper and oxygen interfere. E. HAYES

2098. **Studies on the determination of organic compounds of metals by the extraction method. VIII. Extraction and colorimetric estimation of iron with phenazone.** E. Sudo (*J. Chem. Soc. Japan, Pure Chem. Sect.*, 1954, **75** [9], 968–970).—Ferric iron, NH_4SCN and phenazone form a complex in HCl soln. (pH 2·5) which can be extracted by organic solvents. The experimental conditions for its analytical use were studied. The pink compound can be satisfactorily extracted with a 5-ml portion of ethyl acetate. The extinction coeff. (470 mμ) is proportional to the concn. of Fe within the range 0·2 to 10 μg of Fe per 5 ml of ethyl acetate. Interference results from the presence of Cu, Bi or Co. K. SAITO

2099. **Study of errors in the chemical analysis of steel and cast iron.** A. B. Shaevich (*Zh. Anal. Khim., SSSR*, 1954, **9** [6], 373–376).—Systematic and chance errors in the chemical analysis of standard samples are studied separately. Systematic errors are of the same order as the chance errors of single determinations and exceed the errors due to chance when the average of a number of determinations is obtained. The use of standard samples for control is thus of prime importance. G. S. SMITH

2100. **Analysis of steel by paper chromatography.** G. Venturello and A. M. Ghe (*Ann. Chim., Roma*, 1954, **44** [12], 960–977).—The steel is dissolved in HCl (1 + 1), oxidised with a little HNO_3 and applied to Whatman No. 1 filter-paper. The radial technique is employed, with *n*-butanol - ethyl acetoacetate - conc. HCl (*d* 1·19) (37·5:37·5:25 per cent. by vol.) as developing solvent. To separate Ni and Cr in a sector of the same chromatogram, a second development with *n*-butanol - HSCN is used. The metals are made visible with the usual reagents. A general examination of the intensity and width of the bands permits a rapid classification of the steel. Qualitatively, small quantities ($\simeq$ 0·03 per cent.) of Ni, Cr, Mn, V, Mo, Tl, Co, Cu or Al may be detected in the presence of large amounts of Fe. Quantitatively, by means of standard curves (band width against log percentage concn., and reciprocal of band area, obtained microphotometrically, against log percentage concn.) it is possible to determine Ni, V and Co, within $\pm$ 0·1 per cent. of the normal chemically determined values. L. A. O'NEILL

2101. **Complexiметric titrations (chelatometry). IX. Determination of nickel in the presence of cobalt.** R. Přibil (*Chem. Listy*, 1954, **48** [6], 825–827).—The complex of EDTA (**I**) with Ni reacts readily with KCN with the liberation of an equivalent amount of **I**; the complex of **I** with Co^{III}, obtained by the oxidation of Co^{II} with ammoniacal H_2O_2 in the presence of **I**, is unreactive towards KCN. These differences of behaviour form the basis of a simple complexiметric determination of Ni and Co in the presence of each other. The method is suitable for amounts of Co $>$ 15 mg, for in greater concentrations the blue colour of the complex of Co^{III} with **I** makes the end-point in the subsequent titration with $MgSO_4$ difficult to observe. *Procedure*—To a weakly acidic soln. containing Ni and Co add a known amount of **I** and determine its excess by titration with $MgSO_4$, thus giving the sum of the two metals. After adding ammoniacal H_2O_2 (2 to 3 ml), changing the colour from a wine-red to red - violet, and KCN (1 g), titrate the liberated **I**, after 3 to 5 min., with $MgSO_4$ to the original colour. G. GLASER

2102. **Separation of rhodium and iridium by ion exchange.** M. L. Cluett, S. S. Berman and W. A. E. McBryde (*Analyst*, 1955, **80**, 204–209).—Rhodium and iridium can be separated by passing a soln. of the metals as chloro salts through a column of anion-exchange resin (Amberlite IRA–400). The soln. containing Rh and Ir is added to a 2 per cent. soln. of NaCl in 0·1 *M* HCl containing 5 per cent., by vol., of saturated bromine water; the bromine maintains the Ir in the quadrivalent state. Ir is retained on the column and the Rh is eluted with the same acid NaCl - Br soln. Ir is then eluted with a soln. of 5 *M* aq. NH_3 in *M* NH_4Cl, followed by either 6 *M* HCl or 8 *M* HNO_3. The resin is pretreated by stirring it with a large excess of NaCl soln. and is stored in the same soln. Determination of Rh in the eluate can be made by the $SnCl_2$ procedure of Sandell ("Colorimetric Determination of Traces of Metals," 2nd Ed., Interscience Publishers Inc., New York and London, 1950, p. 523), optical densities being measured at 470 mμ. Small amounts of Ir ($<$ 1 mg) may be determined absorptiometrically after oxidation with $Ce(SO_4)_2$ (Maynes *et al.*, *Anal. Abstr.*, 1954, **1**, 1853) and larger amounts by potentiometric titration (McBryde *et al.*, *Can. J. Res.*, 1950, **22**, 590). A. O. JONES

2103. **Application of paper-chromatographic methods of analysis to geochemical prospecting.** E. C. Hunt, A. A. North and R. A. Wells (*Analyst*, 1955, **80**, 172–194).—The method described consists essentially in applying an aliquot of a suitable extract of a soil to one end of a filter-paper strip and allowing an organic solvent mixture to diffuse upwards through the test spot. The solvent is chosen to produce a separation of the desired metal, which is then detected by spraying the strip with a suitable reagent. The amount of metal present is ascertained by comparison with standard strips. Paper cut to a special design permits ten samples to be dealt with simultaneously. Applications of the method to the determination of Cu, Co, Ni, Nb, Ta, Pb and U are described. A. O. JONES

2104. **Spectrographic determination of contamination of rock samples after grinding with alumina ceramic.** P. R. Barnett, W. P. Huleatt, L. F. Rader and A. T. Myers (*Amer. J. Sci.*, 1955, **253** [2], 121–124).—Samples of quartzite and massive quartz ground between discs of sintered Al_2O_3 (Coors Porcelain Co.) suffered negligible contamination (at worst 0·003 per cent. of Mg; the figure for Al is not stated). The suitable mounting of the plates in a vertical spindle grinder is illustrated.
J. A. SUGDEN

2105. **Application of the logarithmic sector to quantitative spectrographic analysis of petroleum-ash residues.** E. B. Childs and J. A. Kanehann (*Anal. Chem.*, 1955, **27** [2], 222–225).—A well-mixed sample containing $\simeq$ 0·05 g of ash is ignited carefully, the residue is cooled, moistened with H_2SO_4 (1 drop) and after excess of acid has been removed by fuming, the ash is ignited at 1000° F. After accurate weighing, a 0·01-g sample of the residue is mixed with 0·1 g of Li_2CO_3 and 0·001 g of In (as In_2O_3). The mixture is placed in a cratered electrode and examined spectrographically, the spectrograph being equipped with a logarithmic sector disc (full dimensions are given). The resulting spectral lines are triangular in shape with a narrow base; the intensity ratios are measured by the difference in heights of the lines, determined on a graticule. The In 2932·6 A line is taken as internal standard. On 13 samples of various petroleum-oil residues and catalysts, the average standard deviation of Al, Mg, Pb, Sn, Si, Fe, Cr, Ba, Ca, Cu and Ag is $\pm$ 30 per cent., relative to the chemical analytical figures.
D. A. PANTONY

2106. **Polarography of humic acid-like oxidation products of bituminous coal.** A. F. Cody, S. R. Milliken and C. R. Kinney (*Anal. Chem.*, 1955, **27** [3], 362–366).—Because of the reported abnormal properties of the humic acid-like oxidation products of bituminous coal, abnormalities in the polarographic behaviour of humic acids were studied. Three waves appear in the polarogram, and vary normally with variation in drop time, temp., buffer, carrier ions, pH and concn. The first and most persistent wave is believed to be due to the reduction of nitro groups, but the structures responsible for the other two have not been identified. Estimates of the mol. wt. of the humate ions indicate a value for the acids of < 1000.
J. H. WATON

2107. **Analysis for industry.** A. M. G. Macdonald (*Ind. Chem. Mfr*, 1955, **31** [362], 144–145).—Recent methods for the determination of ash, H_2O, CO_2, N, S and P in coal and coke are reviewed.
D. R. PECK

2108. **Suggested guide to metal cleaning [prior to electroplating].** J. C. Harris, W. Stericker and S. Spring (*Bull. A.S.T.M.*, 1955, [204], 31–34).—The proposed scheme for the evaluation of metal cleaners or metal-cleaning methods includes: sources and amount of dirt (oils, grease, wax, rust-preventives, buffing compounds, etc.), prep. of test-panels, cleaning techniques, measurement of dirt removal and reporting of results. The following tests for the determination of metal cleanliness are summarised and their relative sensitivities are listed: water-break (contact angle), water-break (spray pattern), atomiser, fluorescent dye, $CuSO_4$ dip, potassium ferricyanide paper and radioactive tracer. The radioactive tracer and atomiser methods are the most sensitive.
W. J. BAKER

See also Abstracts 2133, 2217, 2218, 2226, 2265, 2275.

3.—ORGANIC ANALYSIS

2109. **Ammonium sulphamate as substitute for lead dioxide in micro-determination of carbon and hydrogen.** A. S. Hussey, J. H. Sorensen and D. D. Deford (*Anal. Chem.*, 1955, **27** [2], 280–281).—Ammonium sulphamate, suspended on silica gel (full preparative details are given), is used instead of PbO_2 in a standard combustion micro-analytical train. No increase of precision or accuracy is claimed over PbO_2 (results for the analysis of 30 samples are given), but the life of the $NH_4SO_3NH_2$ is much longer, and more than 100 analyses can be performed with one filling.
D. A. PANTONY

2110. **Potassium permanganate in the Kjeldahl method for the determination of nitrogen in organic substances.** A. E. Beet (*Nature*, 1955, **175**, 513–514).—The use of an oxidising agent instead of oxygen carriers in Kjeldahl determinations is recommended (*cf. Anal. Abstr.*, 1954, **1**, 2953). The material is digested with H_2SO_4 for 5 min., cooled slightly and $KMnO_4$ is added in small amounts until the charring disappears on shaking. The product is boiled for 1 min., cooled again, and after the addition of sufficient $KMnO_4$ to produce a dirty sage-green colour is digested for a further 1 min. The NH_3 is determined by steam-distillation from the alkalised digest into a three-quarters saturated H_3BO_3 soln., using a silica condenser, and titration of the condensate with 0·01 *N* HCl (methyl red - methyl blue indicator). An individual determination can be completed in 30 min., or 36 in one working day. Satisfactory results have been obtained on the semi-micro scale with coal, cereals, feeding-stuffs, leather and alkaloids, and on the micro scale with pyridinecarboxylic acids, tryptophan and alkaloids.
A. R. ROGERS

2111. **Kjeldahl method with sealed-tube digesti n Factors influencing ammonia decomposition.** B. W. Grunbaum, P. L. Kirk, L. G. Green and C. W. Koch (*Anal. Chem.*, 1955, **27** [3], 384–388).—A study was made of the factors influencing ammonia decomposition in the sealed-tube Kjeldahl digestion procedure. The loss of NH_3 in the digestion is due to its oxidation to N gas and may be caused by (*a*) the decomposition of NH_4HSO_4 to N at temp. exceeding 500° C and (*b*) the oxidation of NH_3 by SO_3 or by O over the same temp. range. The quantity of H_2SO_4 used and prolonged digestion times may also be responsible for considerable loss of NH_3. The data obtained indicate that digestion times of 0·5 hr. are more than adequate for most organic compounds and that addition of a little water to the digestion mixtures markedly increases the stability of NH_3 in H_2SO_4.
G. P. COOK

2112. **Characterisation of organic substances by differential thermal analysis. General experimental technique.** H. Morita and H. M. Rice (*Anal. Chem.*, 1955, **27** [3], 336–339).—Differential thermal analysis was applied to the study of the thermal characteristics of organic liquids and solids. Results showed that the temp. at which a substance exhibits endothermic and exothermic reactions in the

presence of calcined alumina are unique characteristics of that substance, and the resulting thermograms may be used as a means of identification. Experimental procedures and a selection of thermograms of some synthetic and natural high polymers are given in detail. G. P. Cook

2113. **Reproducibility of mounting of solid samples of chlorine-36 compounds for radioactivity measurements.** P. Sorensen (*Anal. Chem.*, 1955, **27** [3], 391–392).—A series of measurements with four different organic ^{36}Cl compounds show that errors introduced in preparing the samples are usually small in comparison with the counting error. G. P. Cook

2114. **Chromatographic determination of acetylene and diacetylene in the presence of monosubstituted acetylenes.** L. Nebbia and B. Pagani (*Chim. e Ind.*, 1955, **37** [3], 200–201).—The chromatographic determination of acetylene (**I**) and diacetylene (**II**) is carried out on a column of Al_2O_3 (96 per cent.) and Cu_2Cl_2 (4 per cent.); 0·1 per cent. of **I** may be detected. Because of partial oxidation of the copper salts, speed is necessary in the determination. Small amounts of **I** and **II** in the presence of vinyl-, methyl-, ethyl- and phenylacetylene and allene may be detected. C. A. Finch

2115. **Statistical comparison of three methods for determining organic peroxides.** C. Ricciuti, J. E. Coleman and C. O. Willits (*Anal. Chem.*, 1955, **27** [3], 405–407).—The three methods compared were the polarographic procedure of Willits *et al.* (*Anal. Chem.*, 1952, **24**, 785), the $SnCl_2$ procedure modified by Barnard and Hargrave (*Anal. Chim. Acta*, 1951, **5**, 476) and the Wheeler iodide procedure (*Oil & Soap*, 1932, **9**, 89). With pure hydroperoxides the three methods are comparable, but with impure products the polarographic technique may give more reliable results. For some samples the chemical methods gave values that were significantly higher than those by the polarographic method. G. P. Cook

2116. **Quantitative determination of alcohols of the aliphatic series by means of ultra-violet colorimetry.** S. A. Shchukarev, S. N. Andreev and I. A. Ostrovskaya (*Zh. Anal. Khim., SSSR*, 1954, **9** [6], 354–358).—Alcohols (methyl, ethyl, *n*-propyl, *iso*propyl, *n*-butyl, *iso*butyl, *n*-octyl or *sec.*-octyl alcohols) in aq. solution are determined by conversion into the nitrites and examination of the absorption in the range 400 to 320 mμ in light petroleum solution. Calibration curves covering the concn. range 10^{-4} to 10^{-3} mole per litre are almost identical for all the alcohols except methyl and permit the total alcohol concn. in a mixture to be determined. G. S. Smith

2117. **Chromatography of organic acids with non-esterifying solvents.** R. W. Scott (*Anal. Chem.*, 1955, **27** [3], 367–369).—Several organic acids were separated by chromatography on silicic acid, with ketones as eluting agents. Good separations were obtained with *iso*butyl methyl ketone - methylene chloride mixtures, different concentrations being required for the various acid constituents. Circular-paper chromatography was used to check the purity and identity of the acid fractions, and was capable of detecting 0·5 to 1 μg of most non-volatile organic acids. The method was applied to the determination of organic acids in plant tissues. G. P. Cook

2118. **Quantitative determination of chloroacetic acids by means of Raman spectra.** M. del Pilar Jorge and J. R. Barceló (*An. Real Soc. Esp. Fis. Quim.*, 1955, **51B** [2], 125–130).—The Raman spectra of mixtures of mono-, di- and tri-chloroacetic acids, (**I**), (**II**) and (**III**), are examined with a Zeiss spectrograph. Linear relationships are found between the log of the ratio of intensities and the log of the ratio of concentrations of pairs of acids, which relationship is not affected by the presence of varying amounts of the third acid. The lines used are 2956(k)K corresponding to **I** and 200(e)K corresponding to **III** in mixtures of **I** with **III**; 783(k)K corresponding to **II** and 277(k)K corresponding to **III** in mixtures of **II** with **III**; 2956(k)K corresponding to **I** and 3016(k)K corresponding to **II** in mixtures of **I** with **II**. The method is suitable only where the proportion of each acid in the mixture is 10 per cent. or higher. D. Leighton

2119. **Spectrophotometry of vinyl acetate in the ultra-violet. I. Determination of acetaldehyde and crotonaldehyde in vinyl acetate.** C. Capitani and E. Milani (*Chim. e Ind.*, 1955, **37** [3], 177–182).—A method for the spectrophotometric determination of acetaldehyde and crotonaldehyde in vinyl acetate is described; it is applicable when the aldehydes are present separately or together. Reference curves of standard solutions of optical density at max. absorption are illustrated, and may be used for comparisons. A formula for use when both aldehydes are present is provided. C. A. Finch

2120. **Structure of reducing disaccharides by lead tetra-acetate oxidation.** A. S. Perlin (*Anal. Chem.*, 1955, **27** [3], 396–399).—The use of lead tetra-acetate oxidation for determining the structure of reducing disaccharides is described, the oxidations requiring only a few mg of material and only 5 or 6 hr. Measurements included the amount of formic acid and formaldehyde produced and the consumption of the lead tetra-acetate. Each position of the glycosidic link was associated with a characteristic oxidation pattern, and results agreed generally with the thesis that reducing sugars were oxidised as the cyclic hemiacetal. The behaviour on oxidation of monomethyl monosaccharides was similar to that of the corresponding disaccharides, allowances being made for the contribution of the non-reducing end-units in the latter compounds. G. P. Cook

2121. **Determination of monomethylamine in mixtures of methylamines.** L. Nebbia and F. Guerrieri (*Chim. e Ind.*, 1955, **37** [3], 198–200).—Methylamine is determined in mixtures containing dimethylamine, trimethylamine and ammonia by precipitation, in neutral solution, of the corresponding Ni dithiocarbamate, after elimination of the Ni dithiocarbamate of dimethylamine in alkaline solution. The determination of methylamine (6 mg) in the presence of dimethylamine (1 g) and ammonia (0·6 to 0·7 g) is described; trimethylamine does not interfere. C. A. Finch

2122. **A mercurimetric method of determination of thiourea.** H. L. Kies (*Z. anal. Chem.*, 1955, **145** [1], 5–9).—The contents of solutions of thiourea or allyl thiourea are determined by standard Hg^{II} solutions by means of the dead-stop titration technique with two mercury electrodes. D. R. Glasson

2123. **Determination of hydroxy and amino compounds by a chlorine-36 isotope-dilution method.** P. Sorensen (*Anal. Chem.*, 1955, **27** [3], 388–390).—A method is described whereby non-chlorine-containing compounds may be analysed by a ^{36}Cl dilution procedure. The compound to be analysed is quant. converted into a chlorine-containing derivative and this is determined by an ordinary isotope-dilution method. For the determination of hydroxy and amino compounds, the 3-chloro-4-methoxybenzoyl derivative was chosen, since radioactive 3-chloro-4-methoxybenzoic acid can be prepared with an acceptably high yield of ^{36}Cl. Percentage recoveries of 94·3 to 101 were obtained in the determination for phenol, 98·6 to 99·5 for catechol, 99·3 to 101·8 for methanol, 98·2 to 99·5 for ethylene glycol, 98·9 to 100·3 for aniline and 82·1 to 95·4 for ethylenediamine. G. P. Cook

2124. **Colorimetric determination of phenol in mixtures of phenol and cresol.** A. I. Kartashevskiĭ (*Neft. Kh.-vo*, 1954, [5], 73–74; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 45,136).—A sample (0·25 to 0·5 g) of the mixture is dissolved in water containing 1 ml of 10 per cent. NaOH soln. and the vol. is made up to 100 ml. A 10-ml aliquot is neutralised to methyl orange with dil. HNO_3 soln. (1 + 4) and diluted to 100 ml. Freshly prepared Millon's reagent (1 ml of Hg dissolved in 10 ml of fuming HNO_3 and diluted with 16 ml of water) (5 ml) is added to 5 ml of this soln. in a test-tube; 5 ml of a standard soln. of phenol (0·025 per cent.) are placed in a second tube and treated in the same way. Both tubes are placed in a bath of boiling water for 30 min. and then rapidly cooled; 5 ml of dil. HNO_3 are added to each tube and the contents are diluted to 100 ml. The colours are compared in a KOL-1 M absorptiometer with a No. 5 filter. The error is < 1 per cent. E. Hayes

2125. **A chromatographic separation of *meta*- and *para*-cresols.** D. White and D. W. Grant (*Nature*, 1955, **175**, 513).—The isomers of cresol ($\simeq$ 100 μg each) may be separated by partition chromatography on a column (45 cm × 1 cm) of "Celite 535" (13 g), with 5 ml of 0·5 *M* phosphate buffer, pH 11·5, as the stationary phase and *cyclo*hexane as the mobile phase. The eluate is collected in 2-ml fractions and analysed colorimetrically with alkaline 2-chloro-4-nitrobenzenediazonium naphthalene-2-sulphonate, which gives a red colour with 5 p.p.m. of cresols. The *o*-isomer is eluted first, then the *p*- and finally the *m*-isomer. A. R. Rogers

2126. **Quantitative analysis of isomers of *cyclo*hexylphenol by means of infra-red absorption spectra.** S. Tanaka (*Japan Analyst*, 1953, **2** [3], 228).—*Ortho*- and *para*-isomers of *cyclo*hexylphenol (an intermediate product for the prep. of 6-*cyclo*hexyl-2:4-dinitrophenol, an agricultural chemical) can be determined in mixtures by the use of their different absorptions of infra-red rays (13·32 μ for *ortho*, 12·13 μ for *para*). The extinctions of both absorption bands are in accord with Beer's law up to 5 mg per ml in CS_2 soln. The standard deviation is $\simeq$ 1 per cent. K. Saito

2127. **Analysis of technical 1-naphthylacetic acid and its methyl ester.** Yu. A. Baskakov and N. N. Mel'nikov (*Khim. Prom-st'*, 1954, [2], 47–49; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 43,521).—A sample of technical methyl 1-naphthylacetate is saponified with KOH soln.; the acid is extracted from the acidified saponification mixture with benzene and determined by adding water to the benzene soln. and titrating with 0·05 *N* KOH soln. at 70° to 75° C. 1:5-Naphthalenediacetic acid is then extracted from the acidified saponification mixture with ether; the ether is removed by distillation, the residue is dissolved in 50 per cent. ethanol, and the acid is determined by titration with 0·05 *N* KOH soln. E. Hayes

2128. **Determination of dibutyl phthalate in propellants.** M. A. C. Mullaly (*Analyst*, 1955, **80**, 237–239).—The method described was developed to avoid the use of ammonium sulphide, which causes certain difficulties in Lamond's method (*Brit. Abstr. C*, 1949, 333). The finely ground cordite containing 1 g of nitroglycerin is extracted for 4 hr. with pure ether in a sintered-glass crucible (grade 3 porosity). The extract containing $\simeq$ 1 g of nitroglycerin, 0·2 g of dibutyl phthalate and 0·3 g of carbamite (diethyldiphenylurea) in a 250-ml flask is freed from ether by a current of air, 10 g of anhyd. $FeCl_2$, 60 ml of methanol, 15 ml of conc. HCl and 15 ml of water are added, and the mixture is heated under reflux on a sand-bath for 45 min., cooled and diluted with 100 ml of water. The dibutyl phthalate is extracted with 150 ml of ether, and the ethereal extract is washed with 100 ml and then with 50 ml of 5 per cent. K_2SO_4 soln. After removal of the solvent by a current of air, the dibutyl phthalate is saponified with 10 ml of 0·4 *N* ethanolic KOH on a sand-bath for 45 min., the liquid is diluted with 50 ml of water and the excess of KOH is back-titrated with 0·1 *N* HCl (thymol blue). A blank determination is made with a synthetic mixture of composition similar to that of the ethereal extract, but without the dibutyl phthalate. The single extraction with ether is sufficient to yield an accuracy of $\pm$ 1 per cent. A. O. Jones

2129. **Polarographic behaviour of some alkyl phthalate esters.** G. C. Whitnack, J. Reinhart and E. St. Clair Gantz (*Anal. Chem.*, 1955, **27** [3], 359–362).—Ethanolic soln. of several alkyl phthalic esters are examined polarographically with tetramethylammonium chloride as supporting electrolyte. Each ester gives 2 well-defined diffusion currents. The effect of pH, height of the Hg column and concn. on the half-wave potential and diffusion current is studied. Measurement of the first wave in neutral or alkaline soln. appears to be suitable for analysis. By using the measured value of the diffusion coeff. for diethyl phthalate, substitution in the Ilkovič equation leads to the number of electrons involved in the reductions, which is four for the first and two for the second. The first reduction product is phthalide, and the difference in potential between the two waves is large enough to permit good yields on large-scale electrolysis. The final reduction product cannot be isolated, but may be 2-hydroxymethylbenzaldehyde. J. H. Waton

2130. **Potentiometric determination of aromatic amines and phenols using chloramine B [with KBr] as a brominating agent.** A. Singh (*J. Indian Chem. Soc.*, 1954, **31** [8], 609–611).—The quant. determination of phenols and aromatic amines by the potentiometric method is carried out with chloramine B in a solution containing KBr and HCl. R. J. Magee

2131. **New gravimetric method for the determination of benzidine.** P. Spacu, M. Brasoveanu and V. Spiridonescu (*Comun. Acad. R.P. Române*, 1953, **3** [5–6], 217–221; *Referativnyĭ Zh.*,

Khim., 1954, Abstr. No. 40,052).—Reinecke's salt $[Cr(NH_3)_2(SCN)_4]NH_4$, precipitates benzidine from HCl or acetic acid soln. as violet-red crystals of $[Cr(NH_3)_2(SCN)_4]_2H_2C_{12}H_{12}N_2$. *Procedure*—Add a ten-fold excess of a freshly prepared soln. of Reinecke's salt to an aq. soln. of benzidine hydrochloride acidified with HCl. After 30 min., collect the ppt. in a crucible and wash it with a soln. containing 0·13 g of Reinecke's salt and 0·125 ml of conc. HCl per 100 ml of water and then with two 1-ml quantities of water. Dry in a vacuum-desiccator and finally in an oven at 105° C. The ppt. is soluble in ethanol and ether. E. HAYES

2132. **Spot reaction for acidic polynitro compounds.** F. Feigl and V. Gentil (*Anal. Chem.*, 1955, **27** [3], 432–433).—Enolisable nitro compounds react with rhodamine B to form red-violet salts, whose red solutions in benzene show orange fluorescence in u.v. light. *Procedure*—One drop of weakly alkaline test soln. is treated with 5 drops of reagent (0·1 per cent. of rhodamine B in 4 per cent. HCl) and the mixture is shaken vigorously with 5 drops of benzene-ether (1:1). A red or pink colour in the upper layer and orange fluorescence signify the presence of enolisable polynitro compounds. G. P. COOK

2133. **Titrations in strongly alkaline media. IX. Titration of hydrazine, isoniazid and hydroxylamine with potassium ferricyanide.** J. Vulterin and J. Zýka (*Chem. Listy*, 1954, **48** [6], 839–842).—Hydrazine, isoniazid and hydroxylamine can be conveniently and accurately determined by direct potentiometric titration with 0·1 *M* $K_3Fe(CN)_6$ in 10 to 25 per cent. KOH. The following reactions occur, respectively: $NH_2NH_2 + 4K_3Fe(CN)_6 + 4KOH \rightarrow 4K_4Fe(CN)_6 + N_2 + 4H_2O$; $C_5H_4NCONHNH_2 + 4K_3Fe(CN)_6 + 4KOH \rightarrow 4K_4Fe(CN)_6 + C_5H_4NCO_2H + N_2 + 3H_2O$; $2NH_2OH + 4K_3Fe(CN)_6 + 4KOH \rightarrow 4K_4Fe(CN)_6 + N_2O + 5H_2O$. With KOH concentrations > 25 per cent. the results are no longer reproducible. G. GLASER

2134. **Action of cyanogen bromide on the pyridine nucleus. Spectrophotometric study.** P. Douzou and A.-M. Le Clerc (*Anal. Chim. Acta*, 1955, **12** [3], 239–247).—An investigation related to previous work (*Brit. Abstr. C*, 1953, 2559) on the spectrophotometric determination of nicotinic acid and amide is reported. The products of the reaction of BrCN with nicotinamide and quinoline, respectively, are isolated. These deriv. have the empirical formulae $C_7H_7O_2N_3$ and $C_{10}H_8ON_2$, and u.v. and i.r. absorption spectra indicate the presence of certain functional groups. W. C. JOHNSON

2135. **Action of periodic acid on piperazine and estimation of piperazine by titration of its precipitated monoperiodate.** A. Wickström and A. Valseth (*Ann. Pharm. Franç.*, 1954, **12** [12], 777–787).—Since the oxidation of piperazine with HIO_4 is not regular, the formation of an insoluble periodate is used to estimate piperazine. To 5 ml of an ethanolic solution of 15 to 40 mg of piperazine are added 5 ml of ether, 1 ml of 0·4 *M* ethanolic hexamine (to prevent formation of the diperiodate) and 1 ml of 0·5 *M* HIO_4. After being set aside for 10 min. at 10° C, the ppt. is filtered off on a Jena (G3) sintered-glass filter, washed with ethanol-ether, dissolved in 10 ml of 5 per cent. H_2SO_4 and diluted to 100 ml. KI (10 per cent.) is added and the liberated iodine is titrated with $Na_2S_2O_3$ (1 mol. of piperazine ≡ 8 atoms of I). E. J. H. BIRCH

2136. **Infra-red functional group analysis of arylsilanes.** M. Margoshes and V. A. Fassel (*Anal. Chem.*, 1955, **27** [3], 351–353).—An infra-red method is described for the determination of the concn. ratio of phenyl and *p*-tolyl substituent groups in tetra-arylsilanes and hexa-aryldisilanes. The absorption maxima for the *p*-tolyl group bands fall within the range 12·47 to 12·50 μ and the phenyl group bands within the ranges 13·40 to 13·45 μ and 14·30 to 14·33 μ. The molar absorptivities of the functional groups are not constant, but accurate results are possible. Certain substituent groups on the Si atom can interfere; aliphatic groups absorb strongly in the 12 to 15-μ region, silanols absorb at 12·4 μ in soln., and the Si–H group gives an absorption band at $\simeq$ 12·5 μ. G. P. COOK

2137. **Determination of unsaturated compounds in the kerosene-gasoline fractions of solid-fuel hydrogenation products.** N. V. Milovidova and B. M. Rapoport (*Tr. Vses. N.-I. In-ta Iskusstv. Zhidk. Topliva i Gaza*, 1954, [6], 137–145; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 45,141).—The electrometric titration method of DuBois and Skoog (*Brit. Abstr. C*, 1948, 254) is used; this depends on the occurrence of a sharp change in the potential between platinum electrodes when an excess of Br is present in soln. The sample (0·2 to 2 g) is dissolved in 110 ml of a mixture of glacial acetic acid, methanol, carbon tetrachloride, 6 *N* H_2SO_4 and 10 per cent. ethanolic soln. of $HgCl_2$ (80 + 7 + 15 + 2 + 2) and is titrated at 0° to 5° C with 0·625 *N* bromate-bromide soln. (17·42 g of $KBrO_3$ + 70 g of KBr per litre). The method is more rapid and simpler than other halogen methods and substitution is reduced to a minimum. E. HAYES

2138. **Rapid method for the determination of non-hydrocarbon gases in commercial liquid fuel gas.** A. E. Heron (*J. Inst. Petrol.*, 1955, **41** [374], 63–66).—The hydrocarbons are absorbed in glacial acetic acid, the major part (90 to 95 per cent.) in a first absorption pipette of a gas-analysis apparatus, and the remainder in a second pipette. The residual gas (mainly N and O) is then washed with $\simeq$ 25 per cent. aq. NaOH to remove acetic acid vapours, and measured over brine acidified with H_2SO_4. The reproducibility of the method is $\pm$ 0·1 per cent. A modified Orsat apparatus and the details of the procedure are described. J. M. JACOBS

2139. **Testing of "L.P.G." [liquefied petroleum gases] and similar hydrocarbon gases. Notes on procedures currently used in the U.K.** The Petroleum Gases Panel of the Institute of Petroleum (*J. Inst. Petrol.*, 1955, **41** [374], 54–62).—Four-fifths of the L.P.G. sold in the U.K. is commercial butane, for which the Liquid Fuel Gas Industry Committee's specification is: total content of C_5 and higher hydrocarbons (determined by low-temp. distillation) $\not>$ 2 mol. per cent. calculated as n-C_5H_{12}; max. v.p. at 45° C, 100 lb per sq. in. abs., as determined by the NGAA (Natural Gasoline Association of America) method; total S $\not>$ 0·02 wt. per cent. (by IP 107); mercaptan S $\not>$ 2·0 grains per 100 cu. ft. at s.t.p. (saturated); H_2S (moist Pb acetate paper) nil; the odour of the gas must be distinctive, unpleasant and non-persistent. The specification for commercial propane is the same except for the total content of C_4 and higher hydrocarbons, which must be $\not>$ 10 mol. per cent., calculated as n-C_4H_{10}, and the max. v.p., at 45° C, 270 lb per sq. in. abs. Total S is determined by IP 107, a lamp method, making use of a special wick-type burner. The products of

combustion are absorbed in aq. Na_2CO_3 and the concn. of S is determined gravimetrically or turbimetrically. Mercaptan S is determined by removing H_2S with acidified $CdSO_4$ and absorbing the mercaptans in 0·2 *N* $AgNO_3$. Formerly, Cd acetate was used for removing the H_2S, but it was found that this reagent could also absorb mercaptans. For the determination of the moisture content of L.P.G., several methods based on the use of the Fischer reagent have been worked out for routine estimations and have been used with good reproducibility. All these methods employ a titration cell (in which the gas passes through the Fischer reagent) followed by a gas meter, with a dry-tube or Hg bubbler trap between the cell and the meter to prevent back-diffusion of moisture from the meter. Another method is based on the principle that when a hygroscopic salt or liquid NH_3 is kept in a dry state between two electrodes no current passes until the salt or NH_3 has taken up sufficient moisture to form a continuous film. Proposed alterations in the methods specified for the determination of v.p. are discussed. J. M. JACOBS

2140. **Purification, purity and freezing points of sixty-four American Petroleum Institute standard and research hydrocarbons.** A. J. Streiff, A. R. Hulme, P. A. Cowie, N. C. Krouskop and F. D. Rossini (*Anal. Chem.*, 1955, **27** [3], 411–415).—The purification and determination of freezing point and purity are described for eleven paraffins, three alkyl*cyclo*propanes, one alkyl*cyclo*pentane, four alkyl*cyclo*hexanes, twenty-four mono-olefins, twelve alkylbenzenes, three dicycloparaffins, three dinuclear aromatics, one cycloparaffin - aromatic and two olefin - cycloparaffins. Freezing points and cryoscopic values are listed. G. P. COOK

2141. **Chromatography of dyestuffs intermediates. I. Paper chromatography of naphthylaminesulphonic acids.** J. Latinák (*Chem. Listy*, 1954, **48** [6], 843–846).—Paper chromatography is suitable for the separation of naphthylaminesulphonic acids (**I**) and for the determination of their purity. Two systems of solvents have been used, *n*-butanol - acetic acid - water (4:1:5) and *n*-butanol - pyridine - water (3:1:1). The R_F values of 19 **I** in each system are listed. The spots are detected either by their fluorescence under u.v. light or by spraying with a soln. of *p*-nitrobenzenediazonium chloride. G. GLASER

2142. **Determination of combined acetic acid content of cellulose acetate. Gravimetric method.** G. Garetto and A. Ruffoni (*Anal. Chem.*, 1955, **27** [3], 400–401).—The method is based on weighing the cellulose regenerated from the cellulose acetate sample by its complete saponification in an aq. alkaline medium. Good agreement with volumetric methods was obtained. G. P. COOK

2143. **Interesting micro-reactions of chemical fibres of proteins and of alginic acid.** W. Bobeth (*Faserforsch. u. Textiltech.*, 1955, **6** [1], 20–25).—The microscopic changes undergone by the various artificial fibres of proteins (casein, groundnut protein, zein) on treatment with conc. H_2SO_4 and by Ca alginate fibres on treatment with 20 per cent. H_2SO_4 are described and illustrated with photomicrographs. These changes may serve to identify the fibres. The protein fibres treated with conc. H_2SO_4 exhibit swelling accompanied by lengthening, with the development of the semblance of a "shining light." With fibres of casein and of groundnut protein, the lengthening proceeds intermittently and produces internal tears in the fibres, with consequent formation of a series of hollow spaces in the fibre core along the length of the fibres. These hollow spaces are rhombic and appear black under the microscope, and serve to distinguish casein- and groundnut-protein fibres from zein fibres, which do not exhibit this effect. On treatment with 20 per cent. H_2SO_4, Ca alginate fibres develop a characteristic microscopic growth of hairs owing to the formation of gypsum crystals. H. L. WHITEHEAD

2144. **A chemical method for the determination of protein rayons in mixtures with wool.** E. Druce (*Shirley Inst. Mem.*, 1955, **28** [4], 67–75).—The chemical properties of Ardil, Fibrolane BX and Vicara are first discussed. A weighed sample of the protein rayon - wool mixture is digested overnight at room temp. with 3 per cent. aq. peracetic acid. The wool, modified by this oxidation, is then dissolved by treatment with 0·1 *N* NaOH at the temp. of the boiling-water bath for 15 min. The residue of protein rayon is washed, dried and weighed. A correction is applied to account for the small amount of protein rayon that dissolves. Results are consistent and accurate to within 1 per cent., but tend to be slightly high because of a small insoluble residue of wool. A. M. SPRATT

2145. **The determination of refrigerating agents in refrigerator oils (Philipp test).** German Standard DIN 51,593 (*Erdöl u Kohle*, 1954, **7** [12], 838–840).—The new standard proposes that the period of test of the stability of the refrigerants in lubricating oils shall be increased to > 96 hr., as compared with the old figure of > 48 hr. The definition of the stability of refrigerants in refrigerator oils is given as the time (in hours) which passes, under the conditions of the test, before the first trace of the acid breakdown products is detectable. The apparatus used and methods of testing are described in detail. C. J. KEATTCH

2146. **Quantitative analysis of urea - formaldehyde condensation products. II.** L. M. Mobers (*Plastica*, 1955, **8** [1], 16–19).—The essentials of nine methods for determining methylolformaldehyde and its dimethylene ether are quoted from the literature. (41 references.) (*Cf. Anal. Abstr.*, 1955, **2**, 1269.) P. S. ARUP

2147. **Reaction of poly(vinyl chloride) with pyridine.** H. Freytag (*Z. anal. Chem.*, 1955, **145** [1], 24–26).—Wechsler's colour reaction of poly(vinyl chloride) with pyridine in the presence of sodium methoxide is investigated. Intermediate formation of glutaconic dialdehyde, as its sodium enolate, is indicated by an intensive red coloration with 2-naphthylamine in acid solution. The pentamethine dyestuff produced is extracted with pentanol and further characterised by its reversible colour change from red to yellow in alkaline and acidic solutions. D. R. GLASSON

2148. **Ultra-violet spectrophotometric determination of styrene and of phthalate and fumarate esters in polyester resins.** R. C. Hitt, R. G. Schmitt and R. W. Stafford (*Anal. Chem.*, 1955, **27** [3], 354–356).—Unsaturated polyester formulations, comprising polymeric esters of dihydric alcohols and dibasic acids dissolved in a vinyl monomer such as styrene, were analysed for phthalate ester and styrene concn. by u.v. spectrophotometry. Measurements were made at 291 and 282 mμ and from the resultant

absorbancies the styrene, phthalate, fumarate and/or maleate ester concn. were calculated.

G. P. Cook

2149. **Measuring viscosity of thermosetting resins by parallel-plate plastometry.** D. I. Marshall (*Bull. A.S.T.M.*, 1955, [204], 40–44).—The parallel-plate plastometer (*Brit. Abstr. A1*, 1946, 373) can be used for measuring viscosities at various temp., and changes of viscosity during thermal hardening, of most thermosetting resins (except melamine-formaldehyde types) having viscosities from 10 to 10^7 poises. The modified apparatus and procedure are fully described; the method is not continuous and is inapplicable to materials departing appreciably from Newtonian behaviour. Desirable features are: use of very thin layers (0·8 mm) of resin, rapid attainment of temp. equilibrium, quick recording of viscosity and no cleaning problem (0·001-in. aluminium-foil separates the resin from the 4-in.-diam. platen). Viscosity - time curves for phenolic, urea and polysiloxane resins are shown and discussed. W. J. Baker

2150. **Gas quality measurement.** H. Mellors (*Gas World*, 1955, **141** [3684], 842–847).—Recent advances in the methods for the measurement of the calorific value, the hydrogen sulphide content and the sp. gr. of town's gas are reviewed. The conditions for obtaining accurate measurements with the Boys, Fairweather, Thomas recording, Sigma and other calorimeters are discussed. A new type of compensator for the Fairweather calorimeter which has reached an advanced state of development obviates the need for a test-room of greater than average height. J. M. Jacobs

See also Abstracts 2105, 2106, 2268.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

2151. **The rapid estimation of whole blood in homogenised tissue preparations.** M. W. Gordon and J. I. Nurnberger (*J. Histochem. Cytochem.*, 1955, **3** [2], 130–133).—The weighed tissue is homogenised and made up to volume, so as to contain about 0·5 per cent. of blood. To an aliquot is added a pinch of saponin and the mixture is centrifuged. The supernatant liquid is divided into two parts, one of which is treated with a stream of illuminating gas for 5 min. and the other with compressed air under the same conditions. The absorption is read at 562 mμ, and the increment due to the formation of carbonyl haemoglobin (Δ) is noted. Then $\frac{\Delta \times V}{0{\cdot}26 \times G}$ = percentage of blood in the tissue, where V is the volume to which the weight, G, of the tissue is diluted. N. E.

2152. **Quantitative determination of urine sodium by means of ion-exchange resins.** J. C. Vanatta and C. C. Cox (*J. Biol. Chem.*, 1955, **212** [2], 599–605).—A modification of the resin method of Vanatta and Cox for the determination of serum sodium (*Anal. Abstr.*, 1955, **2**, 409) is described for the determination of Na in urine; this is necessary because of the higher content of K and NH_3 in urine, and because urine contains a substance that interferes with the bromothymol blue indicator when this is added before the solution is boiled. A column of Amberlite IR-112 (55 cm instead of 50 cm) is used and, after the urine is placed on this, elution is effected with 0·05 *M* $BaCl_2$ at a rate $\geq$ 2 ml per min. The eluate is collected in the 50 to 132·5-ml zone, the Ba is pptd. as $BaSO_4$, and the NaCl in the filtrate is converted into NaOH by passage through a column of Amberlite IRA-400. The resulting solution is boiled for 10 min. to expel NH_3, and the NaOH is then determined by titration with 0·01 *N* HCl, with bromothymol blue indicator.

J. N. Ashley

2153. **Procedure for the determination of sodium, potassium and chloride in biological material.** W. H. Hulet (*Amer. J. Med. Sci.*, 1955, **229** [1], 81–88).—Five grams of homogenised material are digested with 3 ml of HNO_3 (AnalaR) by boiling in a 125-ml Erlenmeyer flask. The digested sample is transferred to a 100-ml calibrated flask, the volume is adjusted with water and the undissolved material is filtered off. Determinations of Na^+ and K^+ are made on the filtrate by flame photometry, using a direct-reading photometer with the Beckman Model DU quartz spectrophotometer. Oxygen and acetylene are used for the flame. Chloride is determined by the $HgNO_3$ method. R. S. Tonks

2154. **Determination of sodium in bone with the aid of cation-exchange chromatography.** G. B. Forbes and M. D'Ambruso (*J. Biol. Chem.*, 1955, **212** [2], 655–661).—A new method is described for the determination of Na in bone. An acid solution of bone ash is passed through a column (1·2 cm × 45 cm) of Dowex 50-X12 (a sulphonated polystyrene resin). Elution with 0·7 *N* HCl gives NaCl (unaccompanied by P, K, Ca or Mg) in the 100 to 300-ml zone of eluate. This is then analysed directly for Na by flame photometry without the need for correction for interfering substances. The elution procedure, which includes reconditioning the column for the next run, requires approx. 7 hr. The method is applicable to 0·3 to 2 g of bone (wet wt.). The recovery of Na from synthetic mixtures is 100 ± 3 per cent., and of Na added to solutions of bone ash is 97 ± 8 per cent. J. N. Ashley

2155. **Studies in histochemistry. XXXII. Flame-photometric determination of potassium in microgram quantities of tissue.** D. Glick, R. H. Swigart, S. N. Nayyar and A. R. Stecklein (*J. Histochem. Cytochem.*, 1955, **3** [1], 6–15).—Water extraction of the potassium from microtome sections of adrenal tissue, from which fat had been removed with petroleum spirit, was shown to be preferable to either digestion or ashing, in which a considerable loss of potassium occurred. A Beckman DU spectrophotometer with flame photometer and photomultiplier attachments was used with a red-sensitive phototube. The aq. solution of the sample was transferred to the sample vessel and the intensity of the emission at 768 mμ with a slit width of 0·62 mm and the 0·1 sensitivity setting was determined. Standards of KCl solution containing 0·01 to 0·10 milliequivalents per litre were used for comparison. N. E.

2156. **Colorimetric estimation of ultramicro-quantities of calcium in human serum as the complex with alizarin.** S. Natelson and R. Penniall (*Anal. Chem.*, 1955, **27** [3], 434–437).—A sample of fresh serum (0·02 ml) is added to 1 ml of water, and 2 ml of *N* triethanolamine (aqueous) and 3 ml of alizarin-octanol reagent are added (40 mg of recryst. alizarin

in 1 litre of *n*-octanol). The mixture is shaken for 20 min. and centrifuged for 5 min. Approx. 2 ml of the upper layer are removed, centrifuged and measured in the Klett - Summerson colorimeter with the 56 filter or at 560 mμ with a spectrophotometer. A calcium standard and a "blank" determination are carried out simultaneously. The Ca can be pptd. as the oxalate from serum or urine before the colorimetric determination. Mg interference in the serum is ≃ 7 per cent., for which a correction can be made; Sr and Ba also interfere. The mean recovery from 11 determinations was 101·5 per cent. and the standard deviation was ± 0·15 mg for a mean of 9·43 mg per 100 ml.

G. P. Cook

2157. **Colorimetric determination of trace quantities of boric acid in biological materials.** W. C. Smith, jun., A. J. Goudie and J. N. Sivertson (*Anal. Chem.*, 1955, **27** [2], 295–297).—The biological fluid (2 ml) containing ≯ 30 μg of boron is added to Li_2CO_3 (0·1 g) in a platinum crucible. The soln. is evaporated (steam-bath) and the residue is heated slowly to 650° C and maintained at that temp. for 1·5 hr. After cooling, the C-free residue is mixed with 6 *N* HCl (2 ml) and the suspension is centrifuged. One ml of the supernatant liquor is treated with conc. H_2SO_4 (5 ml) and 0·025 per cent. carminic acid in conc. H_2SO_4 (5 ml) in a sealed test-tube. After mixing and allowing to stand for 5 min., the absorption is measured at 575 mμ. Boron concn. is calculated from a standard reference curve, allowance being made for a reagent blank. Other substances commonly encountered in biological fluids, NO_3' and NO_2' do not interfere. An accuracy of ± 1 μg is claimed.

D. A. Pantony

2158. **The determination of iron in plasma or serum.** T. H. Bothwell and B. Mallett (*Biochem. J.*, 1955, **59** [4], 599–602).—A simple and rapid modification of previous techniques is described for the determination of Fe in plasma. It is based on the effective separation of Fe from β-globulin (with which it is associated as a complex) in plasma by successive treatment with dil. HCl and trichloroacetic acid. *Procedure*—Add 2 *N* HCl (2 ml) to the plasma or serum (4 ml) and stir with a glass rod. Add 2 ml of trichloroacetic acid (20 per cent. w/v) and stir vigorously for ≮ 45 sec. Centrifuge at 2500 r.p.m. for 20 min., add the supernatant fluid (5 ml) to a mixture of thioglycollic acid (2 drops), 2:2′-dipyridyl (0·5 ml of solution prepared by dissolving 0·4 g in 5 ml of acetic acid and making up to 100 ml with H_2O) and saturated aq. Na acetate (2·5 ml), and shake thoroughly. Determine the colour intensity in an absorptiometer with a 520-mμ filter, and compare with the intensities of solutions of known Fe content.

J. N. Ashley

2159. **Micro-determination of cobalt in biological materials.** B. E. Saltzman (*Anal. Chem.*, 1955, **27** [2], 284–287).—Biological samples are either ignited at 700° C or wet-ashed with HNO_3, the temp. finally being raised to 400° C. The white residue from either method is dissolved in dil. HNO_3 (1 + 1) and the soln. is evaporated slowly to dryness (1 to 2 hr. on the steam-bath). The residue is dissolved either in dil. H_3PO_4 (1 + 49) (25 ml) or water (25 ml) with additions (0·25 ml each) of HNO_3. After cooling, the pH is adjusted to 3 to 4 with purified 50 per cent. hydrated sodium citrate (methyl orange). 1 per cent. 1-nitroso-2-naphthol in dil. acetic acid (1 + 1) (5 ml) is added and, after 1 hr., the Co complex is extracted with 3 × 10-ml portions of $CHCl_3$. The extract is purified by shaking with dil. HCl (1 + 99) (25 ml) and is then evaporated to dryness (steam-bath). The residue is dissolved in HNO_3 (0·5 ml) and to the soln. is added 10 per cent. Na_2SO_4 (1 ml). After evaporation to dryness, the mixed residue is heated (400° C final temp.) with successive 0·5-ml portions of conc. HNO_3 until a white ash is obtained. This is dissolved in dil. H_3PO_4 (1 + 49) (5 ml) and to the soln. is added 0·1 per cent. aq. nitroso-R salt (1 ml) and 50 per cent. Na acetate trihydrate (2 ml). The soln. is digested (steam-bath) for 3 min. exactly, 0·75 ml of conc. HCl is added and the heating is continued for exactly 2 min. After cooling, the soln. is made up to 10 ml, and the absorption measured at 530 mμ against distilled water. The Co concn. is calculated from a standard calibration curve. Cu^{++}, Fe^{++}, Fe^{+++} and Ni^{++} do not interfere seriously. Results are given for the determinations of Co in six animal tissues.

D. A. Pantony

2160. **Determination of glucose in blood by a colorimetric method.** H. Romanovski (*Polski Tygod. Lekar.*, 1954, **9** [6], 182–183; *Referativnŷĭ Zh., Khim.*, 1954, Abstr. No. 43,071).—The method is based on the reaction of glucose with conc. H_2SO_4 to form hydroxymethylfurfural. *Procedure*—To 0·1 ml of whole blood add 1·9 ml of a mixture containing 5 g of trichloroacetic acid and 0·1 g of Ag_2SO_4 in 100 ml of water. Centrifuge or filter and add 3 ml of H_2SO_4 (sp. gr. 1·84) to 1 ml of the filtrate. Heat on a bath of boiling water for 6·5 min., cool and measure the colour spectrophotometrically. The intensity is proportional to the glucose content of the blood.

E. Hayes

2161. **A method for the simultaneous determination of urea and citrulline.** P. Elodi (*Acta Physiol. Acad. Sci. Hung.*, 1954, **6**, 225–233).—Addition of phenylhydrazine to Fearon's diacetyl reaction for urea and citrulline changes the colour of the citrulline condensation product to give a weak maximum absorption between 520 and 540 mμ. To 2 ml of test solution add 1 ml of an acid mixture consisting of equal parts of conc. sulphuric and phosphoric acids, followed by 0·15 ml of 3 per cent. diacetyl monoxime solution and 0·15 ml of aq. 0·0145 per cent. phenylhydrazine hydrochloride. Mix the contents well and immerse in a boiling-water bath for 10 min. Cool and read the extinction values in a spectrophotometer at 495 and 525 mμ against a blank reagent test. The value for each component can then be calculated from their respective calibration curves at these wavelengths.

E. Kawerau

2162. **The quantitative determination of hippuric acid in urine as a test of liver function.** K. Schwandt (*Dtsch. ApothZtg*, 1955, **95** [15], 352–354).—A number of methods for determining hippuric acid (**I**) are discussed and the following method is proposed. Boil the urine, filter hot and make up to the original weight with H_2O. To 5 ml of this solution add hypobromite solution (**II**) (10 ml of Br, 100 ml of 15 per cent. aq. NaOH and 200 ml of H_2O) dropwise, until gas is no longer evolved. Add a further 15 ml of **II**, heat for 30 seconds on a boiling-water bath and cool. Prepare a blank by subjecting a further 5 ml of the solution to the same process but without adding **II**. Extract both lots with ether. Compare the colour with a scale prepared from ferric acetate which has been calibrated against **I**.

K. J. Gardner

2163. **Studies in carbon tetrachloride poisoning. The detection and estimation of creatine and related compounds.** E. Eden, D. D. Harrison and A. W. Linnane (*Austr. J. Exp. Biol.*, 1954, **32** [3], 333–339).—Creatine is completely removed from tissue by two extractions with perchloric acid. *Procedure*—Grind 0·5 g of liver with 1 ml of 10 per cent. $HClO_4$. Rinse the vessel and pestle with 5 per cent. $HClO_4$, centrifuge, decant the supernatant liquid and re-extract the residue with 0·5 ml of 5 per cent. $HClO_4$. Adjust the pooled extracts in an ice-bath to pH 8 (thymol blue) with KOH. Centrifuge to remove the pptd. potassium salt of the acid. Aliquots of the supernatant liquid (0·5 to 5 μg of creatine) are transferred to Whatman No. 1 paper and chromatographed by the descending method in 75 per cent. ethanol for 4 hours. Several strips are run together and on one the creatine spot is revealed by dipping in a reagent consisting of equal parts of 0·05 per cent. aq. diacetyl soln. and 1-naphthol (1 g dissolved in 100 ml of water containing 6 per cent. of NaOH and 16 per cent. of Na_2CO_3). A 5-sq.-cm area centred on the spot is cut out from the replica strip and this is washed for 15 min. with 1 ml of dist. water with occasional shaking. To 0·7 ml of the supernatant liquid 0·2 ml of the 1-naphthol soln. and 0·1 ml of the diacetyl reagent are added and the colour is read after 30 min. with an Ilford 604 filter with maximum transmission at 520 mμ. The standards must also be run on paper as the paper contains an unknown substance which contributes to the blank value. The developing solvent gave good separation of creatine, arginine, glycocyamine, methylguanidine, glutathione and oxidised glutathione with R_F values 0·50, 0·17, 0·40, 0·77, 0·32 and 0·11, respectively. Methods for arginine and glycocyamine are also given. E. Kawerau

2164. **Placental and blood histaminase in human pregnancy.** [**Determination of histaminase.**] T. E. T. Bradshaw and W. J. E. Jessop (*Biochem. J.*, 1955, **59** [4], 603–605).—When certain precautions are taken (*e.g.*, the use of scrupulously clean apparatus), the method of Kapeller - Adler (*Brit. Abstr. C*, 1951, 222) gives consistent results in the determination of histaminase in pregnancy serum and in placental extracts. Attempts to develop a colorimetric modification or to use the NH_3 evolved as a basis for determination are unsuccessful. J. N. Ashley

2165. **A method for the determination of glutamine in cerebrospinal fluid and the results in hepatic coma.** T. P. Whitehead and S. R. F. Whittaker (*J. Clin. Pathol.*, 1955, **8** [1], 81–84).—To 1 ml of cerebrospinal fluid in a 12-ml test-tube add 0·2 ml of 10 per cent. v/v H_2SO_4, place in a boiling-water bath for exactly 10 min., remove and cool in cold water. Add 0·3 ml of 10 per cent. w/v NaOH, 5 ml of water, 0·5 ml of 2 per cent. gum ghatti soln. and 2 ml of Nessler's reagent; read the colour with a blue - green filter. Treat a standard soln. of glutamine (25 mg per 100 ml) similarly. An unheated blank may also be run, but is unnecessary if the fluid is examined on the day of collection. Urea is hydrolysed under the test conditions to the extent of 1 per cent. and a correction is made by determining the urea and deducting 0·05 × concn. of urea found from the total glutamine figure. In normal subjects (18) the glutamine ranged from 6 to 14, in liver cirrhosis (9) from 16 to 31, and in hepatic coma (7) from 30 to 54 mg per 100 ml. The test is recommended in the differential diagnosis of hepatic coma. H. F. W. Kirkpatrick

2166. **The fractionation and determination of corticosteroids in urine.** E. R. Cook, B. Dell and D. J. Wareham (*Analyst*, 1955, **80**, 215–225).—Corticosteroids may be fractionated by a simple partition technique on silicic acid columns impregnated with a controlled amount of water. The ether-washed silicic acid is adjusted to contain 6 per cent. of water and is transferred into the chromatograph tube with benzene saturated with water. The mixed corticosteroids (1 to 4 mg) in 2 to 5 ml of wet benzene are applied to the column, which is eluted serially with 120-ml portions of four benzene - ether mixtures (13 + 2; 9 + 2; 6 + 2; 1 + 1) and finally with 100 ml of ether. The eluate is collected in 10-ml aliquots in an automatic fraction cutter (Grant *et al.*, *Chem. & Ind.*, 1951, [12], 230). The fractions are evaporated to dryness at 90° C. Two absorptiometric methods for determining the separated corticosteroids are described, one based on the procedure of Saffran *et al.* (*Endocrinology*, 1952, **50**, 639) and the other on the colour reaction with phenylhydrazine and H_2SO_4 (Porter *et al.*, *Brit. Abstr. C*, 1951, 215). The method has been applied to the fractionation of extracts of adrenal tissue and of urine hydrolysed with acid and also with β-glucuronidase preparations. A modified procedure with much smaller silicic acid columns is described for the determination of total 17-hydroxy-corticosteroids in urine extracts. The average excretion of these, liberated by crude β-glucuronidase preparations, is 3·5 mg per 24 hr. (males).

A. O. Jones

2167. **A rapid micro-method for the determination of 17-hydroxy- and 17-keto-steroids.** H. Wilson and R. Fairbanks (*Arch. Biochem. Biophys.*, 1955, **54** [2], 440–456).—In the method described, oxidation with CrO_3 is combined with a modification of the micro-assay for 17-ketosteroids described earlier (*Anal. Abstr.*, 1955, **2**, 423). The procedure, which requires 2 to 20 μg of steroid material, is applicable to chromatographic fractions of extracts of biological material and to purified preparations. Most 17-hydroxysteroids of both the C_{19} and C_{21} series react similarly, all except certain 20-methylsteroids among 21 compounds tested giving comparable colours. *Reagent* (**I**)—Reflux glacial acetic acid for 1 hr. over CrO_3, then distil. Immediately before use dissolve 0·2 per cent. of CrO_3 in the purified acetic acid. *Procedure*—Calibrate with duplicate aliquots of a standard soln. of androsterone (0·25 mg per ml in ethanol) containing 5, 10, 15 and 20 μg pipetted into Pyrex-glass tubes (10 mm × 75 mm). Pipette duplicate aliquots of the test soln. containing 2 to 20 μg of 17-ketosteroids into similar tubes. Allow the solvent to evaporate from all tubes and leave overnight in a vacuum-desiccator containing P_2O_5. Pipette 0·02 ml of **I** into each tube, rotate to dissolve the residue and place in a water bath at 40° C for 1 hr. Add 0·10 ml of ethanolic 2 per cent. *m*-dinitrobenzene and complete the determination as described previously, but with 0·30 ml of stabilized ethanolic 2·5 *N* KOH and allowing the reaction to proceed for 90 min. at 0° C. Measure the colour at 530 mμ and plot a calibration graph from the means of the duplicate androsterone standards. Extrapolate to zero androsterone and subtract the intercept value obtained from all readings on test solutions. The corrected optical density is then proportional to the 17-hydroxy- and 17-keto-steroid content as androsterone. W. H. C. Shaw

2168. **The determination of urinary 17-trioxysteroids [17 : 21-dihydroxy-20-ketosteroids].** R. Rivoire, J. Rivoire and J. Poujol (*J. Biol. Chem.*, 1955, **213** [1], 11–18).—A very simple method is described for the determination of the "17-trioxysteroids" (cortisone-like steroids) in urine. After hydrolysis with glucuronidase, the urine is treated with HCl to pH 1, and the steroids are extracted with $CHCl_3$. The residue left after evaporation of the $CHCl_3$ is treated with phenylhydrazine hydrochloride in methanol - conc. HCl. After keeping at 37° C for 1 hr., the optical density is determined at 415 mμ. A control is also carried out and the concn. of "trioxysteroids" is ascertained from a reference graph prepared from tetrahydrocortisone.

J. N. Ashley

2169. **The estimation of hydroxysteroids.** B. Baggett, L. L. Engel and L. L. Fielding (*J. Biol. Chem.*, 1955, **213** [1], 87–97).—A simple and rapid method is described for the determination of hydroxyl groups that can be acetylated with acetic anhydride in pyridine at 100° C for 1 hr. The method has no intrinsic specificity for steroid alcohols, but has been used for the determination of those steroids with hydroxyl groups in the positions and with the configurations usually present in urinary steroids. The method depends on conversion of the acetates into acethydroxamic acids with alkaline NH_2OH, formation of the purple complex with Fe^{+++}, and determination of the intensity photometrically at 560 mμ. Cholesterol is used as a control and a formula is given for calculation of the amount of hydroxysteroid. J. N. Ashley

2170. **Method for the chromatographic separation of very polar steroids.** M. M. Pechet (*Science*, 1955, **121** [3132], 39–40).—A method previously described (*Anal. Abstr.*, 1954, **1**, 1942) is adapted for the rapid separation at room temp. of very polar steroids, in particular cortisone, hydrocortisone and their tetrahydro- and dihydro-derivatives, by prior impregnation of the filter-paper with the saturated aq. phase of the solvent mixture. The mobilities of the steroids relative to that of cortisone in six solvent systems are tabulated. H. F. W. Kirkpatrick

2171. **A micro-method for the detection and assay of steroid C_{21} 17-hydroxy-α-glycols.** H. Wilson and R. Fairbanks (*Arch. Biochem. Biophys.*, 1955, **54** [2], 457–466).—In the method given, C_{21} 17-hydroxy-α-glycols are oxidized with HIO_4 to 17-ketosteroids, which are then determined by the micro-assay described previously (*Anal. Abstr.*, 1955, **2**, 423). Both 17:20:21-triols and 17:20-diols are converted into 17-ketosteroids by HIO_4 treatment, but 20:21-α-glycols without 17-hydroxy groups do not react. The technique, which is sensitive to 5 to 10 μg of material, is made specific by preliminary chromatographic separation and is applicable to both the non-acetylated chromatograph fractions and to purified preparations. At least three 17-hydroxy-α-glycols, totalling 526 μg of androsterone equivalent per 24 hr., were found in normal male urine.

W. H. C. Shaw

2172. **Colour reagent for paper chromatography of steroids.** A. Bodánszky and J. Kollonitsch (*Nature*, 1955, **175**, 729–730).—The reagent (aniline hydrogen phthalate) used by Partridge (*Brit. Abstr. C*, 1951, 59) for tracing sugars in chromatograms has been found suitable for steroids, with which yellow, orange or brown spots on a white background are obtained. Substitution of *p*-phenylenediamine for aniline in this reagent increases the sensitivity tenfold, 2 to 3 μg of steroid giving an easily distinguishable spot. The reaction is selective, only steroids containing the Δ^4-3-keto group in the A ring giving a colour. The colour reaction can also be carried out with oxalic acid in place of phthalic acid, or with 1- or 2-naphthylamine in place of aniline. H. F. W. Kirkpatrick

2173. **The determination of total lipids in blood serum.** W. M. Sperry and F. C. Brand (*J. Biol. Chem.*, 1955, **213** [1], 69–76).—A method is described for the direct, gravimetric determination of the unmodified total lipids of serum or plasma. The lipids are extracted with chloroform - methanol and after purification of the extract (two methods of purification are given) it is evaporated to dryness *in vacuo* in N and the residue is weighed. Wt. of residue × 125 gives concn. of lipids in mg per 100 ml. The method, which requires only a small amount of serum, gives accurate results; extraction of the lipids is complete, and they are completely free from non-lipid contaminants, and oxidative degradation is avoided. J. N. Ashley

2174. **Cup-plate assay of serum fibrinolysin.** J. T. M. Dingle and D. P. Page Thomas (*Nature*, 1955, **175**, 728–729).—To a 0·5 per cent. soln. of fibrinogen in sodium barbitone - HCl buffer (pH 8·0) kept in a water bath at 43° C an equal vol. of 4·0 per cent. soln. of agar in buffer, cooled to 53° C, was added; after mixing by gentle stirring, sufficient soln. was poured quickly on to a levelled glass plate bounded by a sealed frame to give a depth of 3·25 to 3·5 mm. When the gel had set, buffered thrombin was applied to its surface [one 10-ml thrombin ampoule (Maw) for a plate area of 1000 sq. cm] and the plate incubated at 37° C for 2 hr., when the gel became opalescent owing to the pptn. of finely divided fibrin. Excess of thrombin was removed, the requisite number of cups were cut with a cork borer 8 mm in diameter and the cups filled with 0·1 ml of enzyme soln. The assay design was that of antibiotic cup-plate assays. The plate was incubated for 18 hr. and measurement of the lysed zones was made by projection on to a ground-glass screen. A linear relationship was observed between the log. of enzyme concn. and the square of the zone radius over a wide range of enzyme concn. Serum antifibrinolysin was estimated by prior incubation at 37° C for 2 hr. of various serum diln. with a standard enzyme preparation.

H. F. W. Kirkpatrick

2175. **Chromatographic separation of polyphenols [in urine].** S. Fiker (*Pracovní Lékař.*, 1953, **5** [4], 206–209; *Referativnŷĭ Zh., Khim.*, 1954, Abstr. No. 43,066).—The urine of healthy subjects and of workers exposed to an atmosphere containing benzene is examined for polyphenols. The urine is hydrolysed by heating on a water bath with H_2SO_4, the polyphenols are extracted with ether and chromatographed on paper by the descending technique with the solvent system benzene - butanol - water (9:1:10). Three reagents are used to develop the spots: (*a*) ammoniacal soln. of $AgNO_3$, (*b*) Pauli's reagent (*p*-diazobenzenesulphonic acid) dissolved in 2 *N* NaOH soln., (*c*) a soln. of phloroglucinol in 2 *N* NaOH soln. E. Hayes

2176. **Determination of quinine in urine in the "tubeless method" of gastric analysis.** R. D. Lewis and A. G. Foord (*Amer. J. Clin. Path.*, 1955, **25** [2], 199–205).—The method involves the administration of a caffeine diuretic followed 1 hour

later by 2 g of quinine-treated ion-exchange resin. Chloroform is used for the extraction of quinine in urine and results in a more complete extraction than other methods. The final determination is based upon comparison of the fluorescence of the quinine in u.v. light with that of standards.
R. S. TONKS

2177. **A method for the indirect manometric determination of the rate of Pantocaine [amethocaine] breakdown in human serum.** E. H. Maier (*Z. Naturforsch.*, 1954, **9b** [8], 556–560).—Amethocaine (**I**) is split in the organism into diethylaminoethanol and butylaminobenzoic acid. It is a strong inhibitor of serum cholinesterase, and hence retards the degradation of acetylcholine. The rate of breakdown of **I** is measured by incubating serum with **I** for different periods of time in a series of flasks, adding the same amount of acetylcholine to each and measuring the rate of CO_2 production in a Warburg apparatus. Maximal rate of CO_2 production occurs in the flask in which incubation has destroyed all the **I**. It is suggested that acetylcholine and **I** compete for the same enzyme.
E. KAWERAU

2178. **Metabolic demethylation of 5-ethyl-3:5-dimethyloxazolidine-2:4-dione (paramethadione, Paradione).** T. C. Butler (*J. Pharmacol. Exp. Therap.*, 1955, **113** [2], 178–185).—A description is included of a method for the determination in plasma of 5-ethyl-5-methyloxazolidine-2:4-dione ("EMO") (**I**), to which paramethadione (**II**) is demethylated *in vivo*. *Procedure*—To 1 ml of oxalated plasma (containing $\gg$ 100 mg of **I** per litre) in a glass stoppered tube add 3 ml of 5 *M* NaH_2PO_4 and 20 ml of ether, previously purified by distillation and washing successively with aq. NaOH, aq. HCl and water. Shake, centrifuge, transfer 15 ml of the ether layer to another tube and shake with 4 ml of 0·05 *M* borate buffer, pH 9, previously saturated with ether. Centrifuge and measure the optical density of the separated lower layer at 215 mμ and at 220 mμ against a blank of borate buffer saturated with ether. Calculate the concn. of **I** from the difference between the two readings. The molar extinction coeff. of the ionic form of **I** is 9×10^3 at 215 mμ and 3×10^3 at 220 mμ. The apparent content of **I** in normal dog and human plasma is < 6 mg per litre. Unchanged **II** is not determined and does not interfere.
W. H. C. SHAW

2179. **Ion-exchange resin indicator compounds [for indicating the pH of the gastric juice without intubation].** Security Trust Co. of Rochester (Brit. Pat. 728,383, Date Appl. 26.2.51; U.S.A. 25.2.50).—The non-toxic indicator compound comprises an ion-exchange complex synthetic polymer resin with which is associated 0·04 to 0·5 millimole of displaceable and readily identifiable ions (quinine cation, tetramethylaminophenothiazinium ion, or 2:4-diamino-4′-ethoxyazobenzene cation) per dose, the millimoles of the indicator ion per gram of ion-exchange resin being less than the total capacity of the resin.
J. M. JACOBS

2180. **A simple method for desalting biological fluids for chromatography.** B. R. Baliga, K. Krishnamurthy, R. Rajagopalan and K. V. Giri (*J. Indian Inst. Sci., A*, 1955, **37** [1], 18–22).—The electrolytic desalting procedure of Consden, Gordon and Martin (*Brit. Abstr. C*, 1948, 61) gives rather poor recoveries for certain amino acids. The desalting procedure described in the present paper gives recoveries of 100 $\pm$ 10 per cent. Copper cannot be removed and forms an intense red band in the ninhydrin-stained chromatogram just above the glutamic acid - threonine band, which does not interfere with the development or identification of other bands. *Procedure*—Evaporate the required amount of urine, hydrolysed or unhydrolysed, at low temperature and reduced pressure. Add to the residue 1 or 2 ml of ethanol (95 per cent. with 0·5 per cent. v/v HCl) and stir. Set aside for 30 min. and stir occasionally. Filter the clear supernatant liquid and wash the residue (3 or 4 times) with 2 ml of 95 per cent. ethanol. The ethanol concn. must be maintained to prevent solution of the salt, and the dish containing the residue and ethanol should be kept in a bell-jar containing a dish of ethanol. Evaporate the filtrate as before and dissolve the residue in a small volume of 0·5 per cent. v/v HCl to facilitate the solution of glutamic acid and cystine. Use this solution for spotting on the chromatographic paper.
K. A. PROCTOR

2181. **The determination of amino acids with ninhydrin.** E. W. Yemm and E. C. Cocking (*Analyst*, 1955, **80**, 209–213).—The colorimetric method for the determination of amino acids based on the reaction with ninhydrin is systematically examined and the conditions for a stoicheiometric reaction for most amino acids have been established. The amino-acid soln. (1 ml containing 0·05 to 5·6 μg of amino N) is mixed with 0·5 ml of a citrate buffer of pH 5 (prep. described) and then with a soln. containing ninhydrin and KCN in 2-methoxyethanol. The mixture is heated for 15 min. at 100° C, cooled and diluted to a suitable vol. with ethanol. The optical density is then determined against a reagent blank at 570 mμ for all amino acids and at 440 mμ for proline and hydroxyproline. The yield of dioxohydrindylidenedioxohydrindamine (**I**), the probable end-product, is quantitative. All the amino acids investigated gave colours equiv. to 100 $\pm$ 1 per cent. of **I** except tyrosine and phenylalanine (89 per cent.), tryptophan (83 per cent.) and lysine (108 per cent.). Proline and hydroxyproline do not yield **I**, but form a yellow end-product with the ninhydrin.
A. O. JONES

2182. **The quantitative paper-chromatographic determination of amino acids.** W. Gerok (*Hoppe-Seyl. Z.*, 1955, **299** [3–4], 112–128).—Essential conditions for the accurate determination of amino acids by paper chromatography are investigated, and the use of the ninhydrin reagent in relation to the distribution of errors, ease of elution and the stability of the coloured complexes is studied. Losses during migration relative to the types of solvent employed and the regions traversed have been determined, together with a procedure for the precise location of individual amino acids on the chromatogram, which does not interfere with any subsequent quantitative colorimetric evaluation of the eluate. A method incorporating these results is described for the separation of amino acids in protein hydrolysate.
G. R. WHALLEY

2183. **The quantitative paper chromatography of amino acids in protein hydrolysates.** E. Kafrányi (*Hoppe-Seyl. Z.*, 1955, **229** [3–4], 129–138).—The accuracy of the quant. paper chromatography of amino acids is examined, and the results show that the two-dimensional method of Boissonnas gives good qual. results, but not quant. owing to variable amino-acid losses. The percentage losses in one-dimensional systems vary between 0·6 and 54 per cent. A new procedure is described for the location of isolated acids, which requires warming the paper

to 60° C, after spraying with naphthaquinone-sulphonic acid, and subsequent u.v. examination. This method shows a greater sensitivity than the ninhydrin technique. Fluorescent trails are observed behind the migrating spots in all cases, due to adsorption, the amount of which is dependent upon the quantity of amino acid applied to the paper. This observation applies to all types of paper chromatography, thus limiting accuracy in quant. work. G. R. WHALLEY

2184. **A modified procedure for the separation of acidic amino compounds using a sulphonated polystyrene resin.** M. K. Hamdy, W. J. Harper and H. H. Weiser (*J. Dairy Sci.*, 1955, **38** [2], 147–154).—In a modification of the method of Moore and Stein (*Brit. Abstr. C*, 1952, 62) the pH of the buffer used for elution from columns of Dowex 50-X12 resin (sodium form) is altered to 4·0 and the concentration to 0·06 *M*. The modified procedure requires much less time and eliminates the need for temp. control during chromatography. In addition to the compounds given previously (*loc. cit.*), serine phosphate and glutamine were separated. W. H. C. SHAW

2185. **A simple buffered solvent system for the two-dimensional paper chromatography of amino acids.** N. Subramanian and M. V. Lakshminarayan Rao (*J. Sci. Ind. Res., India, C*, 1955, **14** [2], 56–58).—The essential details are given for a simple and reproducible procedure, using phenol treated with a pH 1·0 buffer, coupled with butanol - acetic acid - water (4:1:1) solvent in a two-dimensional run, developed for determining the amino-acid make-up of proteins in certain little-known foods. *Procedure*—For the first run the solvent is prepared by adding 7 ml of 0·2 *M* KCl - HCl buffer to 50 ml of phenol, and the path of the amino acids is buffered by suitably dipping the paper (the common Whatman No. 1 sheets) in the same buffer and drying at room temperature. Descending development is employed, the length and period of run being 18 to 20 in. and about 20 hr., respectively, in each direction. Room temperatures (22° to 27° C) are quite suitable. Between and after the two runs the chromatogram is air-dried thoroughly at room temperature. For colour development with ninhydrin, the sheet is dipped in a 0·4 per cent. solution of the reagent in 95 per cent. ethanol, containing 4 per cent. acetic acid, and heated at 60° C for 30 min. A brief exposure of the chromatogram to dilute ammonia vapour, followed by thorough aeration before dipping, or the inclusion of 2 per cent. of collidine in the reagent, ensures complete colour development. G. C. JONES

2186. **An optical method of determining amino acids after paper chromatography.** P. Darmon and D. Faucquembergue (*Ann. Pharm. Franç.*, 1954, **12** [12], 766–777).—A number of amino acids (0·025 to 0·150-μg samples) are chromatographed in two dimensions on Whatman No. 1 paper with butanol - acetic acid - water (4:1:5) and with a solution containing phenol (15 g), *m*-cresol (30 g) and a buffer at pH 8·3 (7·5 ml). The spots so obtained maintain the symmetry of the original spot and after they have been made visible the max. optical density is measured. The revealed spots are cut out and photographed on microfilm by reflected light at the same time as standard-density screens placed on Whatman No. 1 paper, so that an absolute measure of optical density is obtained when the photograph is measured with a microphotometer. Calibration curves are thus obtained that are of permanent value for the same conditions of chromatography. Direct photometry may be used, but photography gives a greater sensitivity and the advantage of a permanent record. The accuracy claimed is within ± 10 per cent. E. J. H. BIRCH

2187. **A new method for revealing amino sugars on electropherograms.** J. D. Romani (*Compt. Rend. Soc. Biol.*, 1954, **148** [11–12], 1069–1071).—Four times the quantity of serum usually taken for protein electrophoresis is applied to the paper. Reveal the amino sugars after separation as follows. Dry the strip at 60° C, immerse for 2 minutes in 0·5 per cent. periodic acid and wash twice with dist. water. Stain the strip for 15 min. in freshly prepared Schiff's reagent, followed by three successive washes in sodium bisulphite [water (200 ml), sodium bisulphite saturated soln. (10 ml) and 8·25 per cent. HCl (10 ml)]. Wash the strip in running water until bands begin to appear, blot and fix in anhydrous acetone for 30 to 60 min. Dry at room temp. or at 60° C. Put the strip into commercial formaldehyde for 15 to 20 min.; this changes the colour of the bands to mauve and differentiates the background colour. Wash in running water, blot, dry in acetone and finally in a current of air. Render translucent and read without a filter in a photoelectric scanner. All mucopolysaccharides of neutral and medium acidity (pH 4·5) are revealed, but those more acid than pH 4 are not. E. KAWERAU

2188. **Determination of histamine as an isotopic derivative.** R. W. Schayer, Y. Kobayashi and R. L. Smiley (with K. Y. T. Wu) (*J. Biol. Chem.*, 1955, **212** [2], 593–598).—A method is described for the determination of histamine in biological samples. It depends on the conversion of histamine into a radioactive derivative with ^{131}I-labelled *p*-iodobenzenesulphonyl chloride ("pipsyl chloride"), and removal of interfering substances by repeated recrystallisation in the presence of added carrier. Recrystallisations are carried out successively from aq. acetone, acetone - ethanol, acetone - toluene and aq. acetone. The radioactivity is invariably constant after the fourth, and often after the third, recrystallisation. A count is performed after the fourth recrystallisation, from which the amount of histamine is calculated. The advantages of the method over existing techniques are (*a*) it is more specific for histamine, and no known tissue constituents interfere, (*b*) purification is effected after the labelled derivative is mixed with carrier, and (*c*) it can be used over a very wide range of concn. of histamine. The method may be useful for determination of any amine which gives a crystalline "pipsyl" derivative. J. N. ASHLEY

2189. **The amounts of glycerophosphoryl esters in some tissues.** G Schmidt, L M. Greenbaum, P. Fallot, A. C. Walker and S. J. Thannhauser (*J. Biol. Chem.*, 1955, **212** [2], 887–895).—A method is described for the determination of glycerophosphoryl esters in tissues. It depends on esters of glycerophosphoric acid being completely resistant to prostatic and intestinal phosphatases, but hydrolysable to α- and β-glycerophosphoric acid by *N* HCl at 100° C (20 min.), whereas other phosphate esters are hydrolysed by the enzymes. A purified tissue filtrate is prepared; part is used for digestion with prostatic phosphatase before, and another part after, acid hydrolysis. The amounts of inorganic P are then determined in each. The difference between the two values gives the P of the glycerophosphoryl esters. J. N. ASHLEY

2190. **Detection of pyrrolidonecarboxylic acid.** N. Ellfolk and R. L. M. Synge (*Biochem. J.*, 1955, **59** [3], 523–526).—Results are given of ionophoresis and partition chromatography of pyrrolidonecarboxylic acid. The acid can be detected on filter-paper by the chlorine - starch iodide reaction of Rydon and Smith (*Brit. Abstr. C*, 1952, 557). Methods for the detection and determination of pyrrolidonecarboxylic acid are discussed, and possible improvements are suggested. Zone-electrotechnique could be adapted to take advantage of the fact that the acid is the nitrogenous compound which migrates most rapidly towards the anode at neutral pH. J. N. ASHLEY

2191. **Interference in the fluorimetric analysis of pyridine nucleotides by certain ions.** J. P. Kring and J. N. Williams, jun. (*J. Biol. Chem.*, 1955, **212** [2], 751–755).—In the presence of Krebs - Ringer phosphate buffer there is a decrease of fluorescence in the analysis of pyridine nucleotides and N-methylnicotinamide by the method of Huff and Perlzweig (*Brit. Abstr. A III*, 1948, 151) as modified by addition of H_2O_2 to oxidise reduced pyridine nucleotides. This effect is due to the presence of Ca^{++} and Mg^{++}; on an equimol. basis Mg^{++} is more effective than Ca^{++}. Ca^{++} acts by interfering with the formation of the fluorescent material, rather than by quenching the fluorescence once it is formed. An internal standard can be used to compensate for this depression of fluorescence. J. N. ASHLEY

2192. **Metabolism of neoplastic tissue. VI. Assay of oxidised and reduced diphosphopyridine nucleotide in normal and neoplastic tissues.** L. A. Jedeikin and S. Weinhouse (*J. Biol. Chem.*, 1955, **213** [1], 271–280).—A method is described for the determination of oxidised and reduced diphosphopyridine nucleotide in normal and neoplastic tissues. The oxidised form is extracted with a hot buffer, pH 5·4, and is then determined spectrophotometrically after reaction with ethanol and alcohol *apo*dehydrogenase. The reduced form is similarly extracted with a hot buffer at pH 8·7, and is then determined in the same manner after reaction with acetaldehyde and the enzyme. The error is ± 5 per cent. when 15 μg of nucleotide are used. J. N. ASHLEY

2193. **Resolution of ribonucleotides by zone electrophoresis.** A. M. Crestfield and F. W. Allen (*Anal. Chem.*, 1955, **27** [3], 424–425).—A rapid resolution of the isomeric ribonucleotides is possible by electrophoresis. The four 3′ isomers are resolved in a sodium formate buffer of pH 3·5 and ionic strength 0·1, as well as in a 0·1 *M* borax soln., under a field of 30 V per cm in 90 min. Each 5′ isomer is separated from the 2′ and 3′ isomers in a 0·1 *M* borax soln., where it forms a borate complex, under a field of 37 V per cm in 90 min. The separation of the 2′ and 3′ isomers may be achieved for both cytidylic and uridylic acids in a PO_4''' buffer of pH 5·8 under a field of 37 V per cm in 150 min., for adenylic acid at a pH > 5·8 at 40 V per cm in 120 min., and for guanylic acid in a formate buffer of pH 3·8 at 38 V per cm in 240 min., all at an ionic strength of 0·1. J. H. WATON

2194. **A sensitive method for the determination of deoxyribonucleic acid in tissues and micro-organisms.** J. M. Webb and H. B. Levy (*J. Biol. Chem.*, 1955, **213** [1], 107–117).—The deoxyribonucleic acid is hydrolysed in trichloroacetic acid solution and the liberated deoxyribose is determined colorimetrically with *p*-nitrophenylhydrazine in alkaline solution after removal of interfering substances by extraction with butyl acetate. *p*-Nitrophenylhydrazine is specific under the given conditions and is more sensitive than the usually employed diphenylamine. J. N. ASHLEY

2195. **Determination of triose phosphates and proposed modifications in the aldolase method of Sibley and Lehninger.** W. S. Beck (*J. Biol. Chem.*, 1955, **212** [2], 847–857).—In the determination of aldolase by the method of Sibley and Lehninger (*Brit. Abstr. C*, 1949, 295) the chromogens derived from the triose phosphate hydrazones after addition of 2:4-dinitrophenylhydrazine and NaOH are identical for glyceraldehyde 3-phosphate and dihydroxyacetone phosphate, and consist essentially of a mixture of methylglyoxal 2:4-dinitrophenylosazone and pyruvic 2:4-dinitrophenylhydrazone. The reported difference in mol. extinction between the derivatives of the two triose phosphates is related to the rate of formation of the osazone and phenylhydrazone. When the chromogen development reaction is interrupted after 10 min., the ratio between the ϵ_M values of the dihydroxyacetone phosphate derivative and the glyceraldehyde 3-phosphate derivative is 1·8; after 1 hr. the ratio is 1·0. By careful timing of the 10-min. reaction, glyceraldehyde 3-phosphate and dihydroxyacetone phosphate can be determined in a mixture, of which the alkali-labile P is known, by the difference in the rate of osazone formation. For the aldolase assay, times of 15 min. and 1 hr. for the enzyme reaction and chromogen development, respectively, are recommended to increase sensitivity and accuracy. This modification allows use of a triose instead of alkali-labile P as a secondary standard. J. N. ASHLEY

2196. **A simple method for the estimation of pepsin in gastric juice.** A. W. Williams (*J. Clin. Pathol.*, 1955, **8** [1], 85).—The method depends upon the conversion of edestin into edeston by pepsin and the pptn. of residual edestin with NaCl. To 1·0 ml of edestin soln. (0·5 g in 150 ml of 0·1 *N* HCl and 350 ml of dist. H_2O, filtered and kept at 0° C) add 1·8 ml of 0·1 *N* HCl and 0·2 ml of pepsin soln. (50 mg in 100 ml of 0·1 *N* HCl kept at 0° C) and incubate at 37° C. At intervals of 2 min. withdraw a few drops into a saturated soln. of NaCl; absence of opalescence indicates complete digestion and usually occurs in 10 to 15 min.; the exact time is noted. Use 0·2 ml of filtered gastric juice in place of the pepsin soln. and incubate for the same time, then add 3 ml of saturated NaCl soln. and compare the opalescence with standard opalescent tubes. The pepsin may then be calculated directly in terms of the standard pepsin or indirectly in terms of edestin digested. If complete digestion occurs with the test juice, it is diluted and the test repeated. The pepsin and edestin soln. should be freshly prepared daily. H. F. W. KIRKPATRICK

2197. **A new method for the determination of the amidase activity of trypsin': kinetics of the hydrolysis of benzoyl-L-arginineamide.** S. A. Bernhard (*Biochem. J.*, 1955, **59** [3], 506–509).—Although formaldehyde alters the chemical structure of trypsin, the activity of the enzyme is unaffected (as determined by esterase activity), and this is the basis of a potentiometric method for following the amidase activity. At the optimum pH (7·5 to 8·0) in the presence of formaldehyde the trypsin-catalysed hydrolysis of amides may be represented as follows: $R{\cdot}CO{\cdot}NH_2 + H_2O \longrightarrow RCO_2^- + NH_4^+$, and $4NH_4^+ + 6CH_2O \longrightarrow (CH_2)_6N_4 + 4H_3O^+ + 2H_2O$, and hence the hydrolysis can be followed directly by determination of the amount of oxonium ions

liberated in the presence of formaldehyde. In the method described, benzoyl-L-argininеamide is used as substrate. J. N. ASHLEY

2198. **Use of finely emulsified fats for the determination of lipase activity.** M. L. Goldman, T. H. Burton and M. M. Ragman (*Food Research*, 1954, **19** [5], 503–514).—Oil-in-water emulsions were prepared in oil concn. > 20 per cent. w/v by means of 2 per cent. w/v of Astec 4135. After homogenisation, the fat content was determined and the emulsion diluted to 20 per cent. w/v. *Procedure*—Into a 250-ml beaker in a water bath at 35° ± 0·01° C were placed the fat emulsion (10 ml), 0·1 *M* phosphate buffer, pH 5·2 (5 ml), and water (30 ml). Glass and calomel electrodes were immersed in the emulsion and the pH was brought to 8·2 at 35° C. A saline suspension (5 ml) containing 5 to 50 mg of the lipase preparation (depending on its potency) was then added and the reaction was carried out for one hour at 35°C with gentle stirring, while aq. 0·1 *N* NaOH was added at 5-min. intervals to maintain the pH at 8·2. The total vol. of 0·1 *N* NaOH was recorded. A control was carried out with the same amount of the lipase preparation after heating in boiling water for 20 min. N. E.

2199. **An assay method for lipoxidase in animal tissue.** D. H. J. Boyd and G. A. Adams (*Canad J. Biochem. Physiol.*, 1955, **33** [2], 191–198).—An assay for lipoxidase in animal tissues is described which eliminates the interference of haem compounds. Catalytic oxidation of linoleate emulsions at pH 9·0 by haem pigments was inhibited by potassium cyanide, whilst lipoxidase activity was relatively unaffected. Extremely low levels of lipoxidase activity could be detected. Application of the method to beef and pork adipose tissue, uncured bacon, cured unsmoked bacon, and rabbit liver, kidney, spleen, heart, brain and lung indicated strongly that lipoxidase was not present in these tissues and that the linoleate oxidation was catalysed by the haem pigments in the extracts.
C. H. WHITTON

2200. **Manometric estimation of rumen urease.** C. N. Huhtauen and L. S. Gale (*J. Bact.*, 1955, **69** [1], 102–103).—The ordinary manometric method for urease at pH 5·0 cannot be used for rumen contents since large amounts of bicarbonate are present and the pH is near neutrality. The filtered fluid was gassed with CO_2 for a few min. and the pH adjusted, if necessary, with 6·9 per cent. aq. $NaHCO_3$ to 6·8 to 7·0. A Warburg apparatus was used at 37° C. The vessels contained 3 ml of the gassed rumen fluid in the main compartment and 0·1 ml of 0·5 *M* urea in the side arm. The vessels were gassed with CO_2 for 10 min. followed by a 10-min. temp.-equilibration period, when the urea was introduced. Endogenous controls of the rumen fluid were run for each determination. The urease activity was indicated as an absorption of CO_2 from the gas phase by the NH_3 produced from the urea, 0·8 mole of CO_2 being absorbed for each mole of urea. Readings were taken at intervals up to 100 min. N. E.

See also Abstracts 2274, 2292.

Drugs

2201. **Microchemical tests for the identification of alkaloids.** E. G. C. Clarke and M. Williams (*J. Pharm. Pharmacol.*, 1955, **7** [4], 255–262).—Descriptions are given of the crystals obtained from 30 different alkaloids with various reagents by using a micro-technique requiring only 0·01 to 1 μg of alkaloid. Modifications of the ammonium vanadate, selenium dioxide, ammonium molybdate, formaldehyde - H_2SO_4 and Vitali's colour tests are described for the identification of ≃ 0·1-μg quantities of alkaloids. A. R. ROGERS

2202. **Electrometric adsorption analysis of strychnine and brucine. Potentiometric chromatography.** B. Waligóra and Z. Bylo (*Byull. Pol'skoĭ Akad. Nauk, Otd.* 3, 1953, **1** [3–4], 139–143; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 43,529).—Strychnine (**I**) and brucine (**II**) are chromatographed on an alumina column with 50 per cent. ethanol and 20 per cent. methanol as solvents. The presence of **I** or **II** in the eluate is indicated by the occurrence of a sharp peak in the curve relating the potential of an antimony micro-electrode (*Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 39,304) to the quantity of the eluate in ml. With both solvents, **II** is displaced before **I**. E. HAYES

2203. **Separation of mixtures of the alkaloids anabasine and lupinine.** V. V. Udovenko, O. I. Granitova and L. A. Vvedenskaya (*Sb. Statel po Obshch. Khimii, Izd-vo Akad. Nauk SSSR, M.-L.*, 1953, **2**, 1124–1126; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 46,372).—The separation of anabasine from anabasine - lupinine mixtures is based on the formation of complex compounds with copper salts; these have the composition: $CuCl_2.An.2HCl$, $Cu(NO_3)_2.4An.4HNO_3$, $CuSO_4.2An.H_2SO_4.10H_2O$ and $CuCl_2.An.HCl$ (An = anabasine). Copper chloride gives the best separation. E. HAYES

2204. **The assay of some alkaloids and glycosides: a supplement of D.A.B. 6 for inclusion in D.A.B. 7.** F. von Bruchhausen and W. Kussner (*Dtsch. ApothZtg*, 1955, **95** [8], 178–182).—A description of methods and prescribed standards is given for the assays of quinidine sulphate, dihydrocodeinone and dihydrocodeine bitartrates, dihydromorphinone and ephedrine hydrochlorides, theophylline-ethylenediamine and rutin. G. R. WHALLEY

2205. **Colorimetric determination of nicotine.** V. Morani and E. Giovannini (*Chim. e Ind.*, 1955, **37** [2], 109–112).—A method for the determination of nicotine based on reaction with 1-fluoro-2:4-dinitrobenzene is developed. Comparisons with the A.O.A.C. method in which tungstosilicic acid is used show good agreement, but the new method is less cumbersome and more rapid; it can be employed both on commercial products and on raw and processed tobacco. The reagent (1 ml) [prepared by dissolving 1-fluoro-2:4-dinitrobenzene (4 g) in ethanol (100 ml)] is added to the alkaloid soln. [1 to 10 mg of nicotine in 100 ml of ethanol of ≯ 40 per cent. ($d^{15} = 0·951$)], with a H_3BO_3 - $Na_2B_4O_7$ soln. of pH 9·5, and the optical density at 520 mμ is measured and compared with a standard curve.
C. A. FINCH

2206. **A note on the titration of caffeine in pharmaceutical preparations.** A. Anastasi, U. Gallo and L. Novacic (*J. Pharm. Pharmacol.*, 1955, **7** [4], 263–267).—Caffeine (150 to 200 mg), alone or in the presence of acidic substances (such as aspirin), may be potentiometrically titrated with 0·1 *N* $HClO_4$ in anhydrous acetic acid by using a mixture of acetic acid (50 ml) and acetic anhydride (4 ml) as solvent. The function of the acetic anhydride is unknown, but it is not dehydration. Superiority over other methods is claimed for precision and simplicity.
A. R. ROGERS

2207. **The fluorimetric determination of noradrenaline.** K. Erne and T. Canbäck (*J. Pharm. Pharmacol.*, 1955, **7** [4], 248–254).—Dilute (0·001 per cent.) aq. soln. of noradrenaline can be assayed fluorimetrically with ± 2 per cent. error after elimination of sulphite (often present as stabiliser) by ion exchange. Procaine and lidocaine do not interfere. *Procedure*—Pass the sample (10 ml) through a column (10 cm × 1 cm) of anion-exchange resin Amberlite IRA-400 or Dowex 2 previously converted into the chloride form with 20 per cent. NaCl soln. Elute with water, collecting 100 ml. To 10 ml of eluate, add ethylenediamine (1·2 ml), 5 *M* HCl (0·8 ml), and a 10 per cent. solution of ammonium molybdate in 5 *M* HNO_3 (2 drops). Heat for 5 min. on a boiling-water bath, cool, saturate with sodium chloride, and extract for 4 min. with *iso*butanol (30 ml). Separate the *iso*butanol layer, filter into a cell, and measure the fluorescence by using a mercury-lamp source with blue primary and yellow secondary filters. Carry out a reagent blank and a recovery expt. simultaneously. A. R. Rogers

2208. **Paper chromatography of cardiac glycosides and aglycones from *Digitalis lanata*.** K. B. Jensen and K. Tennöe (*J. Pharm. Pharmacol.*, 1955, **7** [5], 334–340).—Eighteen glycosides and aglycones of *Digitalis lanata* are separated by chromatography on formamide-impregnated paper, using as eluents saturated soln. of formamide in chloroform, chloroform - benzene (7:3), chloroform - benzene (4:6), and chloroform - acetone (8:2). The substances (≃ 5 μg each) are located on the chromatograms by heating with trichloroacetic acid, either alone or with chloramine, and examining in u.v. light; the glycosides give a coloured fluorescence differing according to whether the aglycone is digitoxigenin, gitoxigenin or digoxigenin. A. R. Rogers

2209. **Infra-red and X-ray diffraction studies of digitonin.** O. H. Gaebler, J. Parsons and W. T. Beher (*Anal. Chem.*, 1955, **27** [3], 441–443).—X-ray investigation of several commercial samples of digitonin reveals three varieties of compound, two crystalline and one poorly crystalline. Material of the latter type becomes much more crystalline after three crystallisations. Although slight differences occur in the infra-red spectrum between 9·2 and 9·7 μ, these do not correspond to the classification into the above three varieties, since different types can give similar spectra. Absorbance at 750 mμ of the colour produced with anthrone, and the pptn. of cholesterol (Liebermann - Burchard reaction) are essentially the same for all the samples of digitonin. J. H. Waton

2210. **Analytical evaluation of kojic acid.** A. Okáč, L. Sommer and G. Rády (*Chem. Listy*, 1954, **48** [6], 828–838).—Kojic acid (**I**) reacts in neutral or weakly acidic soln. with $Fe^{...}$, $UO_2^{..}$ and $Cu^{..}$ with the formation of characteristic coloured complexes. The composition of the complexes of **I** with $Fe^{...}$ (red or orange - red) and $UO_2^{..}$ (orange - red or orange - yellow) was followed photometrically and their molar extinction coefficients and dissociation constants were determined. The copper salt of **I** (pale-green needles) was prepared by pptg. a soln. of copper acetate (3 g) in H_2O (50 ml) with a 1 per cent. soln. of **I**, followed by *M* sodium acetate (1 to 5 ml). G. Glaser

2211. **Identification of some local anaesthetics by paper chromatography.** E. Scheibe (*Wiss. Z. Humboldt-Univ. Berlin Math-naturwiss. Reihe*, 1952–1953, **2** [5], 15–16; *Referativnŷĭ Zh., Khim.* 1954, Abstr. No. 43,532).—R_F values for a number of local anaesthetics on Schleicher and Schüll No. 2043b paper are determined in different solvents. With the solvent system *iso*butanol - 35 per cent. HCl - water (50:17·5:13·5), a mixture of benzocaine (R_F = 0·90), orthocaine (R_F = 0·67), procaine (R_F = 0·14) and amethocaine (R_F = 0·33) can be separated and the constituents can be identified. For development, ultra-violet light, Ehrlich's reagent (2 g of *p*-dimethylaminobenzaldehyde + 30 g of 35 per cent. HCl soln. + 70 g of water) or 0·2 per cent. $NaNO_2$ soln. in 0·1 *N* HCl followed by 0·2 per cent. ethanolic soln. of 1-naphthylamine are used. E. Hayes

2212. **Polarography of barbituric acid derivatives. III. Derivatives of thiobarbituric acid.** P. Zuman (*Chem. Listy*, 1954, **48** [7], 1006–1019).—Thiobarbiturates give rise to an anodic wave which, in buffer solutions and in *M* NaOH, is subject to complex adsorption influences. The character of the individual waves and the effect of substituents in position 5 were studied. Under alkaline conditions the reaction RSH + Hg ⇌ RSHg + e + H^+, characteristic of thio compounds, takes place. The possibility of polarographic analysis of mixtures of 5-alkylthiobarbiturates and identification of individual compounds is discussed. For analytical purposes a 0·1 *M* NaOH soln. is best, for under these conditions the wave is proportional to the concn. up to 7 × 10^{-4} *M*. G. Glaser

2213. **Determination of Largactil [chlorpromazine] in biological fluids. Supplementary note.** P. Dubost and S. Pascal (*Ann. Pharm. Franç.*, 1955, **13** [1], 56–57).—The difficulty that arose with certain specimens of sulphuric acid in the earlier paper (*Anal. Abstr.*, 1954, **1**, 571) may be avoided by adding to the sulphuric acid 50 mg of potassium metabisulphite per litre or 2 per cent. of ethanol. N. E.

2214. **Modified method for the separation of mercury from toxicological materials.** B. Czerwiecki and S. Wisnicki (*Acta Polon. Pharmac.*, 1954, **11** [1], 51–56; *Referativnŷĭ Zh., Khim.*, 1954, Abstr. No. 34,669).—In the analysis of toxicological materials, Hg was separated as HgS after oxidation of the organic matter. When the HgS was dissolved in HNO_3 and HCl or HCl and $KClO_3$ and the soln. was evaporated to dryness, 22·4 per cent. of Hg was lost. When the HgS was dissolved in the cold in 25 per cent. HCl soln. containing $KClO_3$, the loss was 5·1 per cent. When the Hg was separated from the soln. on a copper disc and distilled with iodine by the method of Rubtsov (*Tr. Gos. N.-I. In-ta Sudebnoĭ Meditsinŷ*, 1949, p. 235), the loss was ≃ 2·9 per cent. E. Hayes

2215. **Determination of metallic iron in "Ferrum redactum" with the aid of copper sulphate.** D. Köszegi and G. Kis (*Magyar Kém. Foly.*, 1953, **59** [4], 112–116; *Referativnŷĭ Zh., Khim.*, 1954, Abstr. No. 41,707).—The iron is allowed to react with $CuSO_4$ to produce Cu, and the excess of $CuSO_4$ is then determined iodimetrically. *Procedure*—A weighed sample (≃ 0·3 g) of reduced iron is shaken for 1 to 2 min. with a mixture of 75 ml of 0·1 *N* $CuSO_4$ soln. and 1 ml of 4·2 per cent. H_2SO_4 soln.; after heating on a water bath for 15 min. with frequent shaking, the mixture is made up to 100 ml and the Cu is filtered off. To 50 ml of the filtrate, 20 ml of 20 per cent. H_2SO_4 soln., 3 g of $Na_4P_2O_7$ (to form a complex with the Fe^{III}) and 5 g of KI are added; the liberated iodine is then titrated with 0·1 *N* $Na_2S_2O_3$. The error is ≃ 0·2 per cent. E. Hayes

2216. **The operating characteristics of some official weight-variation tests for tablets.** C. W. Dunnett and R. Crisafio (*J. Pharm. Pharmacol.*, 1955, **7** [5], 314–327).—Assuming a normal distribution of tablet weights, graphs of probability of acceptance against percentage of defective tablets in a batch are given for four weight-variation tests (based on samples of 10, 20, 50 and 100 tablets, respectively) which are official in various pharmacopoeias, and for two unofficial tests (one based on the standard deviation of the observed weights and the other on a two-sample criterion). It is shown that large samples are more efficient than small ones, and the unofficial tests than the official ones for the same sample size. The individual weights of > 8000 tablets from single-, double- and rotary-punch machines were analysed. Although the number of samples having significant skewness and kurtosis was greater than would be expected if they were drawn from normal populations, there did not appear to be a tendency for either coefficient to depart from its expected value in only one direction.
A. R. ROGERS

See also Abstracts 2045, 2133, 2223, 2225, 2252.

Food

2217. **New method for the determination of zinc in food products.** S. I. Gusev and Z. A. Bitovt (*Vopr. Pitaniya*, 1953, **12** [2], 83–85; *Referativnŷĭ Zh., Khim.*, 1954, Abstr. No. 41,696).—A photonephelometric method for the routine determination of small quantities of zinc in food products is based on the reaction of Zn with diantipyrinylmethylmethane hydrochloride in the presence of SCN′ at pH 2 to 2·5 to form $(C_{24}H_{26}O_2N_4)_2H_2.[Zn(SCN)_4]$. Thiourea eliminates the interfering effect of Cu, and quinol or ascorbic acid that of iron. Phosphates or pyrophosphates do not interfere. Zn can be detected in solutions containing as little as 0·09 μg per ml. Application of the method to millet, barley, peas, potatoes, sunflower seeds and tomatoes gave results lying between 12 and 70 p.p.m. of Zn.
E. HAYES

2218. **Colorimetric methods for the determination of iron, phosphorus and calcium in foodstuffs.** M. T. Valdehita and A. Carballido (*An. Bromatologia*, 1954, **6** [4], 437–453).—The procedure recommended for wet oxidation is to heat 1 g (dry wt.) of the sample with 50 ml of HNO_3, added little by little, boiling gently at first, then adding 70 ml of a mixture of HNO_3 and water saturated with NH_4NO_3 (1:2). Boiling is continued until a colourless liquid is obtained, then dilute HCl is added to the hot solution to complete the hydrolysis of HPO_3 to H_3PO_4, and the mixture is cooled and made up to 50 ml. Aliquot portions of the solution are used for the determination of Fe, P and Cr by standard methods.
L. G. L. UNSTEAD-JOSS

2219. **Measurement of refractometric dry substance of sucrose solutions.** D. F. Charles and P. F. Meads (*Anal. Chem.*, 1955, **27** [3], 373–379).—The accuracy and precision of readings for two commercial refractometers have been evaluated for the determination of purity and concn. of liquid sugar products. The standard deviation of the error of observation for the Bausch and Lomb precision sugar refractometer is ± 0·03 per cent. solids and for the Zeiss sugar refractometer ± 0·07 per cent. A small error is indicated in the international scale of refractive indices of sucrose soln. in the sucrose range 50 to 75 per cent., ≃ 0·07 per cent. solids at 66 per cent. solids.
G. P. COOK

2220. **Use of *p*-aminophenol for the detection of sugars on paper chromatograms.** L. Vámosné Vigyázó (*Magyar Kém. Foly.*, 1953, **59** [3], 253–254; *Referativnŷĭ Zh., Khim.*, 1954, Abstr. No. 41,741).—*p*-Aminophenol (0·5 g) is triturated with 2 g of conc. H_3PO_4, 50 ml of 96 per cent. ethanol are added and the soln. is filtered. For the detection of sugars, this reagent is sprayed on the chromatogram, which is then dried at 105° to 110° C. The following are the colours produced by different sugars and the sensitivity limits: glucose, dark brown on a yellow background (8 μg); fructose, lemon-yellow (8 μg); sucrose, brownish-yellow (14 μg); maltose, brownish-yellow (15 μg); and raffinose, bright brown (8 μg). On chromatograms of acid hydrolysates of starch, the reagent detects 6 oligosaccharides which have not been described previously.
E. HAYES

2221. **Determination of the degree of milling in rice. II. Determination of thiamine and phosphorus for processing control.** H. S. R. Desikachar (*Cereal Chem.*, 1955, **32** [1], 78–80).—Approx. 90 per cent. of the thiamine in rice flour is extracted by shaking for 10 min. with dil. acid, and the same percentage of total P is released by digestion for 5 min. with conc. H_2SO_4. For processing control in rice mills, adoption of short-period extraction and digestion procedures would save time.
S. C. JOLLY

2222. **Sources of error in microbiological determinations of amino acids on acid hydrolysates. II. Apparent loss of amino acids on storage.** M. J. Horn, A. E. Blum, C. E. F. Gersdorff and H. W. Warren (*Cereal Chem.*, 1955, **32** [1], 64–70).—If acid hydrolysates of raw and cooked barley are not filtered efficiently and assayed immediately, certain amino acids disappear from hydrolysates stored at room temp. at pH 6·8 under toluene. The loss varies for different amino acids, depending on the food. Filtration at pH 4·0 removes the material responsible for the loss, and satisfactory recoveries of amino acids can be made from carbohydrate-containing foods, acid-hydrolysed separately and together.
S. C. JOLLY

2223. **Quantitative determination of antibiotics in milk.** L. R. Mattick (*Dissert. Abstr.*, 1955, **15** [1], 1–2).—A method, based on the inhibition of the reduction of NO_3' to NO_2' by *Micrococcus pyogenes* var. *aureus*, is described for the determination of antibiotics in milk. The NO_2' produced is determined colorimetrically after diazotisation of sulphanilic acid and coupling with 1-naphthylamine. The optimum concn. for the determination are: chlortetracycline 0 to 2·0, streptomycin > 1·0 and oxytetracycline 0 to 3·0 μg per ml. Penicillin is detectable at a concn. of 0·1 unit per ml. Bacitracin can also be determined.
S. C. JOLLY

2224. **Determination by flame photometry of the sodium and potassium contents of milk of diverse origin.** M. Ortega (*An. Bromatologia*, 1954, **6** [4], 423–427).—Little variation was found in the potassium and sodium contents of genuine samples of cows' milk; the normal levels were 177 mg of K and 31·2 mg of Na per 100 ml. Human milk gave average values of 46·8 mg of K and 20·7 mg of Na per 100 ml, with variations from 42·5 to 52·9 mg of K and 15·1 to 26·8 mg of Na per 100 ml. The values for asses milk were 64·4 mg of K and 44·4 mg of Na per 100 ml.
L. G. L. UNSTEAD-JOSS

2225. **Aluminium in cows' milk.** J. G. Archibald (*J. Dairy Sci.*, 1955, **38** [2], 159–162).—The photometric method of Hillebrand *et al.* ("Applied Inorganic Analysis," 2nd Ed., John Wiley & Sons Inc., New York, 1953, p. 511) is found to give satisfactory recovery of known amounts of Al added to milk. Control samples gave an average value of 0·46 mg of Al per litre; this was raised to 0·81 mg per litre when the equivalent of 114 mg of Al was added to the daily diet. W. H. C. SHAW

2226. **A quantitative spectrographic method for the determination of tin, copper, iron and lead in milk and milk products. The effect of storage on the concentration of these metals in evaporated milk.** C. W. Gehrke, C. V. Runyon and E. E. Pickett (*J. Dairy Sci.*, 1954, **37** [12], 1401–1408).—In the method described, 5 g of evaporated milk weighed into a Vitreosil crucible are dried at 80°C and then ashed in an electric furnace at 400° C overnight. The thoroughly mixed ash (40 mg) is intimately mixed with 260 mg of a mixture containing 200 mg of spectroscopically pure graphite, 10 mg each of CdO and NiO (internal standards), 20 mg of KCl and 20 mg of $CaCO_3$. Standards are prepared by mixing 200 mg of graphite and 10 mg each of NiO and CdO with 80 mg of a mixture containing 1·00 to 0·0001 per cent. of CuO, Fe_2O_3, PbO or SnO_2 in a mixture of equal parts of milk ash and buffer mixture ($CaCO_3$ and KCl, 1:1). On photographs of the d.c. arc spectra of standards and samples the percentage transmissions are measured by means of a microphotometer at 2840 and 3330·6 A for Sn, 2833·1 A for Pb, 3247·5 A for Cu and 3020·6 A for Fe. The Ni line at 3064·6 A is used as standard for the Fe, the Cd line at 3261·1 A for the remainder. The precision of the method varies from ± 5 to ± 8 per cent. for the four metals. The average Sn content of evaporated milk in electrolytically plated cans was found to increase from 20 to 97·4 p.p.m. after 50 days' storage at 37°C and to 215 p.p.m. after 340 days. The Fe content increased from 6·5 to 16·5 p.p.m. in 340 days; the Cu and Pb contents remained unchanged at 0·68 and 0·35 p.p.m., respectively. W. H. C. SHAW

2227. **The Gerber method of fat estimation. I. Influence of temperature and acid concentration on fat estimation in cream.** F. Kiermeier and G. Pirner (*Z. LebensmittUntersuch.*, 1955, **100** [2], 135–143).—The influence of the reaction temp. on the butyrometric estimation of the fat content of cream is dependent on the method of heating, *e.g.*, by air-bath or water bath. Data are presented on experimental series comparing fat contents determined by the Gerber method with temp. of water bath at 5° C intervals between 60° and 85° C with fat contents determined by the Röse - Gottlieb method. The deviations from the Röse - Gottlieb results are considered. The influence of the concn. of the H_2SO_4 on the reaction is also discussed. The recommendation of Hostettler and Lehmann to carry out the decomposition at 65° C and with 68·7 per cent. of H_2SO_4 appears to present the least disadvantages. S.C.I. ABSTR.

2228. **Photometric estimation of phosphoric acid in the determination of egg content [in foodstuffs].** O. Winkler (*Z. LebensmittUntersuch.*, 1955, **100** [2], 111–114).—The alcohol extract of the food sample is treated according to the method of E. Lindemann, with 5 ml of conc. H_2SO_4, HNO_3 and H_2O_2 soln. (30 per cent.). The HNO_3 and H_2O_2 are driven off and the cooled liquid is placed in a 100-ml calibrated flask, water is added and the vol. is made up to the mark when the soln. is cold. To determine H_2SO_4, 10 ml of the soln. are titrated against *N* NaOH (methyl orange). To estimate H_3PO_4, 50 ml of the soln. are used. A blank is prepared containing 5 *N* H_2SO_4, the amount corresponding with the titration with *N* NaOH (between 10 and 20 ml), and about 40 ml of water are added. To each solution are added 10 ml of ammonium vanadate solution and 10 ml of ammonium molybdate solution, the soln. being well mixed after each addition; the solutions are each made up to 100 ml, shaken and examined at 410 mμ, or with filter S 42 or S 45. S.C.I. ABSTR.

2229. **Determination of cocoa husk in cocoa powder.** Ilona Gál (*Elelmezési Ipar*, 1953, **7** [10], 308–309; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 45,754).—A sample of cocoa powder (3 g) is extracted with 30 ml of ether; 75 ml of 70 per cent. methanol and 5 ml of conc. HNO_3 are added to the residue and, after being heated for 30 min., the mixture is filtered. The residue is washed with hot water, ground and mixed with 16 ml of water and 0·5 ml of a 1 per cent. soln. of methylene blue. A drop of the suspension is examined microscopically in a Burker chamber (magnification × 360) and the number of spirals (scleroidal cells) and total visible particles are counted. The percentage of cocoa husk = $a/0{\cdot}32b$, where a is the total number of spirals, b is the total number of visible particles and 0·32 is a factor established for four different kinds of ground cocoa beans. The method is applicable to cocoa powder of any degree of fineness and also when the cocoa-husk content is less than one per cent. E. HAYES

2230. **Estimation of wort and beer carbohydrates. A simplified method for determining the total fermentable sugars in wort.** W. D. McFarlane, H. R. Held and G. Blinoff (*Amer. Soc. Brew. Chem., Proc. Ann. Mtg*, 1954, 121–127).—The procedure described for the quant. estimation of the carbohydrates in wort and beer (McFarlane and Held, *Ibid.*, 1953, 67), based on the application of the anthrone reaction for colorimetric measurement of carbohydrates separated by chromatography, has been simplified for the estimation of total fermentable sugars in wort. The wort is chromatographed by the above method, but a 0·01-ml aliquot is used instead of 0·02 ml. The chromatogram is cut so that the upper portion contains the fermentable sugars up to and including maltotriose, and the lower portion the dextrins; the vol. of the eluates are 100 ml and 25 ml, respectively. The colorimetric analysis is modified by the introduction of a glucose standard, comprising duplicate tubes each with 4 ml of a standard solution containing 10 μg of glucose per ml. As a check, 1 ml of wort is diluted to 100 ml with distilled water; 1 ml of this solution is diluted to 100 ml and finally a 4-ml aliquot is analysed by the anthrone reaction. In the acetone method, wort is diluted to sp. gr. ≃ 1·015. A 5-ml aliquot is accurately measured into a 50-ml centrifuge tube, 20 ml of redistilled acetone are added at room temp., and the tube is corked and centrifuged for 5 min. at 1800 r.p.m. A 3-ml aliquot of the supernatant liquor is diluted to 100 ml with distilled water. A second dilution (3:100) is made and 4 ml of this solution are transferred by pipette into a colorimeter tube and the anthrone colorimetry is applied. S.C.I. ABSTR.

2231. **Determination of volatile sulphur compounds. II. Further notes on hydrogen sulphide in beer.** M. W. Brenner, J. L. Owades and R. Golyzniak (*Amer. Soc. Brew. Chem., Proc. Ann. Mtg*, 1954, 81–87).—The method (Brenner *et al., Ibid.*, 1953, 83) for the determination of H_2S in beer has been simplified and made more sensitive. Three modifications were made: (*a*) the H_2S is swept out and into the Zn acetate trap at room temp. (instead of at 85° C); (*b*) the quantity of HCl used is increased to 240 ml of 1 + 1 acid (from 100 ml of 7 per cent. HCl); (*c*) the H_2S is absorbed in 20 ml of 2 per cent. Zn acetate (in place of 40 ml) and the final vol. of solution, for development of the methylene blue colour, is 25 ml (instead of 50 ml). Zn acetate soln. (20 ml of 2 per cent.), is placed in a 50-ml graduated cylinder. A drop of silicone anti-foam is placed in a 1-litre flask, the sample of beer (12 fl. oz) is added, followed by 240 ml of HCl (1 + 1). A stream of CO_2 is passed through the beer for 1 hr., the exit gases being passed through glass tubing and through the Zn acetate solution. The cylinder is cooled to 10° C, 2·5 ml of *p*-aminodimethylaniline sulphate solution and 0·5 ml of $FeCl_3$ solution are added and the vol. is made up to 25 ml with 2 per cent. Zn acetate. The liquids are mixed and the optical density is determined at 745 mμ. S.C.I. Abstr.

2232. **Determination of volatile sulphur compounds. III. Determination of mercaptans [in beer].** M. W. Brenner, J. L. Owades, M. Gutcho and R. Golyzniak (*Amer. Soc. Brew. Chem., Proc. Ann. Mtg*, 1954, 88–97).—A method for the determination of microgram quantities of mercaptan S is described. The sample of beer is poured into a 1-litre flask containing a drop of silicone anti-foam, 5 ml of ascorbic acid solution, 2 ml of *o*-phenanthroline and 4 ml of Versene mixture (3 g of Versene acid and 5 ml of Versene T soln. diluted to 50 ml). The solution is distilled and the distillate (200 ml) is divided into two equal portions and to each, transferred to a gas-washing bottle, is added 2 ml of buffer (pH 7). Colloidal S (10 ml) is added to one bottle, then both are placed in a water bath at 30° C and swept with N for 1 hr. The exit gases are bubbled into a graduated cylinder which contains 20 ml of 2 per cent. Zn acetate solution. The receivers are cooled and treated with *p*-aminodimethylaniline sulphate and $FeCl_3$ soln. and the H_2S is determined as in the abstract above (*Anal. Abstr.*, 1955, **2**, 2231). Two mol. of mercaptan are required to form one mol. of H_2S, a factor which decreases the sensitivity of the method. S.C.I. Abstr.

2233. **A short survey of recent methods for determination of "humulones" and lupulone in hops.** M. Meilgaard and A. B. Moltke (*Brauerei Wissenschaft. Beil.*, 1955, [3], 36–37).—The recent advances in hop chemistry due to the development of the counter-current distribution technique as a satisfactory method for the determination of hop bitter principles are discussed. Samples of hops were analysed by the following methods, and the results compared and criticised. (*i*) *Counter-current distribution analysis.* The determination of humulone, cohumulone and adhumulone takes 8 hours, and of total humulones and lupulone 6 hours. (*ii*) *The Govaert - Verzele method.* A benzene suspension of ground hops is passed down a column of specially prepared silica gel, humulones are determined polarimetrically and lupulone by a potentiometric titration. (*iii*) *Lewis's method.* The hops are extracted by procedure (*i*), and the humulones and lupulones are determined spectrophotometrically. (*iv*) *B.I.R.F. method.* Ground hops are extracted with methanol, acidified with HCl (0·2 *N*) and extracted three times with light petroleum. The humulones are determined polarimetrically. (*v*) *CCl_4 method.* Meilgaard has shown that in non-polar solvents, such as CCl_4, the humulones extracted from the hops can be determined by direct polarimetric readings. (*vi*) *Wollmer method.* Humulones are determined by pptn. with lead acetate. This method is useless for old hops. For the determination of lupulone, method (*iii*) gives satisfactory results, while method (*ii*) gives higher values. For the determination of "humulones" in new hops, methods (*ii*), (*iii*) and (*vi*) are at present the only simple reliable methods.

G. H. Baker

2234. **Analysis of condiments.** L. Villanua, M Nuñez Samper, A. Portoles and M. J. F. Pizarro (*An. Bromatologia*, 1954, **6** [4], 399–421).—The method of Scholl and Stroehecker (azeotropic distillation) is recommended for moisture determination, and the method of Sharrer and Kuerschuer for crude fibre. Conventional methods are used for ash and ether extract. For pimento, the method recommended for detecting added artificial colour depends on the addition of 2 vol. of HNO_3 (sp. gr. 1·4) to 1 vol. of a 95 per cent. ethanol extract of the material. The product is stirred for 2 min., then poured into 150 ml of water. A red colour indicates the presence, and a yellow colour the absence, of artificial colour. Methods are given for the microscopical identification of pimento, anise, cinnamon, clove, laurel leaves, table mustard, pepper, thyme and vanilla.

L. G. L. Unstead-Joss

2235. **Colorimetric method for the determination of thiamine in industrial preparations.** O. S. Sherman and S. M. Kogan (*Tr. Vses. N.-I. Vitaminnogo In-ta*, 1953, **4**, 230–234; *Referativnŷĭ Zh., Khim.*, 1954, Abstr. No. 45,160).—In an alkaline medium thiamine (**I**) reacts with diazotised *p*-aminoacetophenone (**II**) to form a coloured compound, which can be measured absorptiometrically. **I** is separated from biological materials by shaking an aqueous extract at pH 2 to 4·5 with white Chapanatinsky clay, which adsorbs 90 to 95 per cent. of **I**. The adsorbate is washed with ethanol and ether and dried at 70° to 80° C. **II** is diazotised at 0° to 6° C by stirring a solution (0·159 g of **II** + 2·25 ml of HCl soln., sp. gr. 1·19, + water to 25 ml) with an equal volume of 4·5 per cent. $NaNO_2$ soln. for 10 min.; four times its vol. of $NaNO_2$ soln. is then added to the mixture and it is set aside for 20 min. To determine **I**, 0·5 ml of the diazotised soln. of **II** is mixed with 2 ml of a soln. containing 2 per cent. of NaOH and 2·88 per cent. of $NaHCO_3$ and, when the rose colour has disappeared (1 to 1·5 min.), the mixture is poured into a cylinder containing 0·1 to 0·2 g of adsorbate (3 to 25 μg of **I**), 1 ml of water and 3 ml of 0·5 per cent. ethanolic soln. of phenol. After mixing for 20 to 30 min., 2 ml of xylene are added and the mixture is shaken for 1·5 to 2 hr. The intensity of the colour in the xylene layer is compared with standards prepared from cryst. **I**, the amount of **I** in these standards increasing in steps of 2 μg. For polyvitamin preparations, the vitamin C is first oxidised. Five tablets are treated with 250 ml of water containing 0·5 ml of 1 per cent. HCl soln. A 1 per cent. $KMnO_4$ soln. is added to 25 ml of this soln. until a rose colour persists; the soln. is decolorised with 0·3

per cent. H_2O_2 soln., diluted to 50 ml with water and filtered. One ml of the filtrate is used for the analysis. The results agree with those obtained by the thiochrome method. E. HAYES

2236. **Riboflavin analysis in foods by a microbiological and chemical (lumiflavin) method.** A. P. Jansen (*Int. Rev. Vit. Res.*, 1955, **25** [4], 365–379).—For the microbiological determination the basal medium of Snell and Strong, as modified by Barton-Wright, was used, the test organism being *Lactobacillus casei*. The method was based on a semi-micro scale (2 ml of final medium; range of the standard curves 5 to 70 mμg of riboflavin). The lumiflavin method depends on the photolysis of riboflavin to lumiflavin in the presence of air. Two methods were used to extract the riboflavin: autoclaving with HCl, and extraction with acid methanol. About 50 foods were analysed. The edible portion of the sample (100 g, or less) was homogenised in a Waring Blendor. After autoclaving with 200 ml of 0·1 *N* HCl at 120° C or extraction with acid methanol (120 ml of methanol, 40 ml of 0·1 *N* H_2SO_4 and 40 ml of H_2O) by refluxing during one hour, the pH was adjusted to 4·5, the liquid was filtered and the vol. made up to 250 ml. For yeast the coeff. of variation were: lumiflavin method, acid extraction, 4·3, methanolic extraction 3·7; microbiological method, acid extraction 7·1, methanolic extraction 7·6 per cent. S.C.I. ABSTR.

2237. **Quantitative biological evaluation of vitamin chromatograms.** G. Marten (*Int. Rev. Vit. Res.*, 1955, **25** [4], 392–401).—A method is described which overcomes the difficulty of measuring the irregular spots on an ascending or descending vitamin chromatogram. Regular circular growth zones may be maintained by means of ring chromatography. The method of making exact measurements is described and illustrated. A Petri dish 30 cm in diameter contains within it a smaller one, or glass ring, on which a round filter-paper is placed, and inside that a very small dish containing a paper wick and the solvent. The test substance in suitable concentration is placed on the round filter-paper with a fine micro-pipette, 4 to 6 of these starting points being placed near the centre on a semicircle. The chromatogram is chemically developed, and radial strips 3 to 4 mm in breadth are cut out and laid on a test medium. After incubation, the growth zones are measured and evaluated by known methods, either graphically or by mathematical calculation. S.C.I. ABSTR.

See also Abstracts 2073, 2075, 2134, 2185, 2198, 2199, 2245, 2263.

Sanitation

2238. **Colorimetric determination of silica [in natural water].** I. Iwasaki, T. Tarutani, K. Katsura and H. Shimojima (*Japan Analyst*, 1953, **2** [3], 210–214).—The colorimetric determination of Si in water was examined from the aspect of the ageing of SiO_2. The pH value at which the sample soln. is stored until analysis, and the acidity at which the analysis is carried out were studied, as well as the influence of the amount of molybdenum reagent, the temp. and the standard soln. The recovery of the reactivity of SiO_2 in natural water, even if treated with alkali, is not always satisfactory. K. SAITO

2239. **A new turbidimetric determination of a small amount of chloride in water.** Y. Kitano and H. Tsubota (*J. Chem. Soc. Japan, Pure Chem. Sect.*, 1954, **75** [9], 931–933).—The turbid, colloidal soln. of AgCl is markedly stabilised by the presence of Pb^{++} and the turbidimetric analysis of a micro-amount of Cl' in natural water is very much improved by the use of a mixed reagent of Ag^+ and Pb^{++}. The reagent (2 ml), containing 0·001 mole of $AgNO_3$ and 0·02 mole of $Pb(NO_3)_2$ per litre, is added to the sample soln. (20 ml containing 0·01 to 3·0 mg of Cl' per litre) and set aside in the dark for 20 min. The turbidity of the soln. is compared with a standard series in Nessler tubes. K. SAITO

2240. **Determination of nitrate in pond water. I.** H. L. Golterman (*Proc. Koninkl. Ned. Akad. Wetenschap., B*, 1955, **58** [2], 109–117).—Methods of determining nitrates in solution by their interaction with either phenoldisulphonic acid (**I**) or 2:4-xylenol are shown to be inaccurate when applied to the determination of small concentrations of NO_3' in pond water, owing to inherent colour and the presence of interfering substances and in particular Cl'. Experiments are quoted which show the effect of Cl' on the determination in which **I** is used, and the steps necessary for their removal, but the method is considered unsatisfactory. H. B. HEATH

2241. **Determination of nitrate in pond water. II.** H. L. Golterman (*Proc. Koninkl. Ned. Akad. Wetenschap, B*, 1955, **58** [2], 118–129).—Detailed investigation of the xylenol method (*Anal. Abstr.*, 1955, **2**, 2240) for the determination of nitrates shows that when modified it can be applied to their determination in pond water. *Procedure*—One drop of H_2O_2 (30 per cent.) and 1 drop of H_2SO_4 (62·5 per cent.) are added to 25 ml of the sample, which is then shaken with 250 mg of Ag_2SO_4. The ppt. is filtered off and washed with a 0·07 per cent. aq. soln. of Ag_2SO_4. The filtrate is made up to 50 ml with the same soln., and 25 ml are transferred to a column containing cation-exchange resin IMAC C 12, which is washed through with water to remove all nitrate. After the addition of a drop of 10 *N* KOH, the soln is evaporated to dryness. The residue is dissolved in 10 ml of H_2SO_4 (62·5 per cent.) and shaken with 1 drop of 2:4-xylenol; 25 ml of benzene and 25 ml of water are added and the whole is transferred to a separator. After being shaken, the bottom layer is withdrawn and shaken with a further 10 ml of benzene. The benzene extracts are bulked and shaken with 4 ml of 2 *N* NaOH and 11 ml of water, 4 ml of 2 *N* NaOH and 5 ml of water and, finally, 5 ml of water. The aqueous fractions are bulked and made up to 30 ml. The absorption is read at 450 mμ against a blank. NO_3' interfere and should be determined separately. The method is accurate to ± 1·5 per cent. H. B. HEATH

2242. **The dichromate method for determining oxidisable organic matter in fresh waters.** E. A. Nikolaeva (*Gidrokhim. Materialy*, 1953, **20**, 68–78; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 42,852).—A sample of water containing 5 to 40 mg of organic matter is evaporated to dryness at 60° to 70° C and the residue is heated to boiling for 5 min. on a sand-bath with 10 ml of 0·4 *N* sulphuric - chromic acid mixture (20 g of $K_2Cr_2O_7$ + 500 ml of water + 500 ml of conc. H_2SO_4) and 100 mg of Ag_2SO_4. The cooled mixture is diluted with water (1 + 5) and transferred to a large flask with a further 100 to 150 ml of water. The excess of dichromate is titrated with ferrous ammonium sulphate soln. with the addition of 2 ml of H_3PO_4 and diphenylamine indicator. A blank experiment is also carried out.

Glucose, tartaric acid, Na oxalate, glycine, alanine and aspartic acid are oxidised almost completely. The results of duplicate determinations are within 3 per cent. of the mean values. A micro-method is used for samples containing less than 2 mg of organic matter. E. HAYES

2243. **Molybdenum blue reaction and determination of phosphorus in waters containing arsenic, silicon and germanium.** H. Levine, J. J. Rowe and F. S. Grimaldi (*Anal. Chem.*, 1955, **27** [2], 258–262).—Factors influencing the determination of P in the presence of As, Si and Ge by the molybdenum blue method are examined and the following procedure is recommended for sea water. The water (100 ml) is evaporated to dryness (steam-bath) and the residue is ignited at 600° C (30 min.). After cooling, the ash is dissolved in HCl (1 + 1) (6 ml) and warm water (35 ml), and to the soln. is added 0·95 per cent. $AlCl_3.6H_2O$ soln. (10 ml). The soln. is boiled and made just alkaline with aq. NH_3 (methyl red) and, after digestion, is centrifuged. The ppt. is separated, washed with 0·1 per cent. NH_4Cl and evaporated to dryness with a little conc. HCl and 46 per cent. HF (1 ml) (steam-bath) and then with conc. HCl (5 ml). The residue is dissolved in HCl (1 to 2 ml) and water (5 ml), and then evaporated to fumes of SO_3 with 48 per cent. HBr (1 ml) and conc. H_2SO_4 (0·2 ml). After cooling, the residue is dissolved in HCl (2 ml) and warm water (≃ 30 ml). To this is added 2 per cent. aq. $(NH_4)_6Mo_7O_{24}.4H_2O$ (5 ml) and 0·5 per cent. $SnCl_2.2H_2O$ in dil. HCl (0·3 ml) and the mixture is made up to 50 ml with water. Light absorption is measured within 30 min. at 735 mμ and the P concn. is calculated from standards, allowance being made for a blank. Results are presented for synthetic samples and for 5 samples of sea water. D. A. PANTONY

2244. **Determination of anionic detergents in sewage, sewage effluents and river water.** J. Longwell and W. D. Maniece (*Analyst*, 1955, **80**, 167–171).—The method described for the determination of anionic detergents in sewage and sewage effluents differs from similar published methods in that extraction with chloroform of the complex of the detergent with methylene blue is made in alkaline soln. and the extracts are washed with an acid methylene blue soln. The aq. soln. of the detergent (100 ml, containing 20 to 150 μg of anion-active material) is mixed with 10 ml of an alkaline phosphate soln. (10 g of aq. Na_2HPO_4 soln. adjusted to pH 10 with NaOH and diluted to 1 litre) and 5 ml of neutral methylene blue soln. (0·35 g per litre) and shaken gently with 15 ml of chloroform. The separated chloroform extract together with 2 ml of chloroform used for washing is shaken with 110 ml of water and 5 ml of acid methylene blue soln. (0·35 g per litre containing 6·5 ml of H_2SO_4). The extract is then filtered through cotton-wool and the extraction of the original soln. is repeated twice in the same manner with 10-ml portions of chloroform. The vol. of the combined extracts is adjusted to 50 ml with chloroform and the optical density is measured at 650 mμ or with an Ilford 607 orange filter. The calibration graph is made by subjecting suitable volumes of a dil. soln. (0·1 g per litre) of sodium dioctylsulphosuccinate (Manoxol O.T.) to the same treatment. This detergent is suggested as a suitable reference standard as it is pure, stable and readily available. A. O. JONES

2245. **Determination of DDT [dicophane] in food products and on various surfaces.** S. V. Zhuravlev and T. P. Kazakova (*Gigiena i Sanitariya*, 1954, [2], 33–37; *Referativnyĭ Zh., Khim.*, Abstr. No. 43,524).—Coarsely ground wheat (100 g) is extracted for 6 hr. with CCl_4 in a Soxhlet extractor. The extract is shaken with 2 × 70-ml quantities of a 10 per cent. soln. of Na_2SO_4 in conc. H_2SO_4 (sp. gr. 1·84), 2 × 70-ml quantities of a mixture of conc. H_2SO_4 and fuming H_2SO_4 (20 per cent. SO_3) (1 + 1) and again with 70 ml of Na_2SO_4 - H_2SO_4 soln. The CCl_4 is distilled off and the residue is nitrated at 100° C for 30 to 45 min. with 4 ml of a mixture of H_2SO_4 (sp. gr. 1·84) and HNO_3 (sp. gr. 1·50 to 1·52) (1 + 1); the cooled reaction mixture is poured into 25 ml of ice-water and extracted with 1 × 25 ml and 1 × 15 ml of ether. The ethereal extract is washed with 25 ml of water, 15 ml of 5 per cent. aqueous NaOH soln. and finally with saturated NaCl soln. After removal of the ether, the residue is dissolved in 25 ml of benzene; 2 ml of *N* ethanolic KOH soln. are added to 5 ml of the benzene soln. and the colour is compared with that produced in standards containing 0·1 to 0·9 mg of dicophane. Modifications of the method are described for the determination of dicophane in milk and in scrapings of plastered and painted surfaces. E. HAYES

2246. **Analysis of pyrethrins. I. Errors arising during the examination of partially degraded materials.** N. C. Brown and R. F. Phipers (*Pyrethrum Post*, 1955, **3** [4], 23–26).—The use of a prefatory chromatographic purification procedure in the analysis of pyrethrins in pyrethrum extract is discussed (*cf. Ibid.*, 1954, **3** [3], 3) in an attempt to remove discrepancies between results obtained on clarified extracts by the Seil method (*Soap*, 1947, **23** [9], 131) and the spectrophotometric method described by Shukis *et al.* (*Soap*, 1952, **27** [11], 124). The instability of the pyrethrins towards artificial light was studied (*cf. J. Sci. Food Agric.*, 1952, **3** [5], 224).—Films of pyrethrinoid material, formed by allowing the solvent of a light-petroleum solution to volatilise from a Petri dish in darkness for 16 hr. at 30° C, then irradiated for three days by, *e.g.*, placing 18 in. below two 300-W tungsten-filament lamps, were divided into fractions soluble and insoluble in light petroleum and analysed by the Seil and spectrophotometric methods. Results of the two methods showed no agreement. Products causing the discrepancy may be (*a*) esters of true chrysanthemum acids and degraded "pyrethrolone," or (*b*) degraded chrysanthemum acids and true pyrethrins or (*c*) esters degraded in both portions of the molecule. S.C.I. ABSTR.

2247. **Comparison of chemical and biological assays of three samples of pyrethrum flowers from Tanganyika.** E. A. Parkin (*Pyrethrum Post*, 1955, **3** [4], 18–22).—Flowers from three strains of pyrethrum grown in southern Tanganyika were compared for pyrethrin content by chemical assay and for insecticidal activity by several techniques of biological assay. The chemical assays were performed on 7 per cent. w/v solutions in Shell oil (P.31) using the 1952 modification of the 1950 A.O.A.C. method. Pyrethrin contents of the three strains expressed as percentages w/w of the dry, powdered flowers were: for sample *A*, pyrethrin I, 0·97; II, 0·84; total 1·82; for sample *B*, I, 0·91; II, 0·54; total 1·46; for sample *C*, I, 0·81; II, 0·63; total 1·44. Biological assays included the tests of powders and of solutions in Shell oil (P. 31) against grain weevils, of the solutions against flour beetles, of wt. loss of flour beetles confined on filter-papers treated with the oil solutions, and the kill of house-flies exposed in a spray chamber to a mist of kerosene

solutions of the extracts. Considerable differences were found between the results of assay.

S. C. I. ABSTR.

See also Abstract 2060.

Agriculture and Plant Biochemistry

2248. **Micro-determination of fluorine in plant material.** R. Fabre, R. Truhaut and A. Rouquette (*Compt. Rend.*, 1955, **240** [2], 226–229).—The finely chopped sample (5 to 10 g) in a platinum or nickel crucible is mixed with a 10 per cent. aq. suspension of MgO (10 ml) and a 10 per cent. aq. solution of Mg acetate (20 ml). After slow evaporation, and drying at 100° C, the residue is ignited for 1 hr. at $\geq$ 600° C in an electric furnace. The ash is treated with conc. H_2SO_4 (or $HClO_4$) plus SiO_2 and the HF is removed by distillation in superheated steam, the entire operation being conducted at const. temp. (146° C) by surrounding the flask with the vapour of boiling tetrachloroethane. The distillate is absorbed in 0·1 *N* NaOH, and an aliquot (50 ml) is acidified with 0·1 *N* HCl and then titrated at pH 3·5 (sodium chloroacetate buffer) with 2×10^{-4} *M* aq. thorium nitrate, with the use of sodium alizarinsulphonate as internal indicator. The colour of the solution changes to rose when formation of thorium tetrafluoride is complete. A blank is run on the MgO and Mg acetate. The all-glass distillation apparatus is shown, and fluorine contents (0·0004 to 0·025 per cent) of cereals grown in different soils are reported. Results are claimed to be accurate to within a few μg. W. J. BAKER

2249. **Estimation of keto acids in plants.** M. Alfthan and A. I. Virtanen (*Acta Chem. Scand.*, 1955, **9** [1], 186–188).—A method is described for determining keto acids in plants. Their 2:4-dinitrophenylhydrazones are prepared and then reduced by tin in ethanolic hydrochloric acid solution to the amino acids, which are investigated by paper chromatography. Quantitative aspects of the method are discussed. C. H. WHITTON

2250. **Determining respiration rate and sampling for chemical analysis of sugar beets.** M. Stout (*J. Agric. Food Chem.*, 1954, **2** [26], 1324–1328).—A sampling technique is described that, without undue injury to the beet, enables respiration rate to be measured and the preparation of diffusate for chemical analysis. The method for respiration rate is similar to that of Nelson and Oldemeyer (*Proc. Amer. Soc. Sugar Beet Technol.*, 1952, 400). Results by a modified colorimetric Stanek - Pavlas method for "harmful nitrogen" correlate well with total and acid-soluble N in fresh beets. S. C. JOLLY

2251. **The quantitative determination of mustard-oil glucosides with the anthrone reagent. VII.** O. E. Schultz and R. Gmelin (*Z. Naturforsch.*, 1954, **9b** [1], 27–29).—Free carbohydrates and other interfering substances are removed from the plant extracts by preliminary chromatography on paper or on columns of anionotropic alumina. The anthrone reagent in conc. sulphuric acid can then be used for the determination of the glucosides, as the constituent glucose is liberated during the reaction. From the glucose value, the amount of glucoside can be obtained by calculation. Good agreement was found between estimated and actual values. E. KAWERAU

2252. **Determination of penicillin in feeding-stuffs.** R. Brunner and H. Margreiter (*Öst. Chem-Ztg*, 1955, **56**, 36–39).—Fodder containing an admixture of 2 to 10 g of antibiotics per kg was suspended in butyl acetate and buffered to pH 2. After centrifuging, 50 ml of the clear fraction were treated with 20 ml of buffer solution of pH 7·2. The contents of penicillin in the aqueous layer were determined iodimetrically and biologically. The method is claimed to be simple and accurate. Results, summarised in three tables, provide evidence for the stability of procaine penicillin in preparations of feeding-stuffs. S.C.I. ABSTR.

See also Abstracts 2057, 2127.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

2253. **Apparatus for quantitative absorption of gases.** B. F. Ormont and V. I. Smirnova (*Zh. Anal. Khim., SSSR*, 1954, **9** [6], 364–365).—Three types of absorption tubes for gases are described, one of which is suitable for ultramicrogravimetric titration. G. S. SMITH

2254. **General-purpose analytical absorption column.** D. Hausding (*Z. anal. Chem.*, 1955, **145** [1], 1–5).—An analytical absorption column for gases or readily volatile liquids is described. A reflux water-condenser and three absorption traps are set vertically in series, facilities being provided for subsequent titration of their contents. Applications described in detail include the determination of NH_3 in fertilisers or from the Kjeldahl method, sulphites in the presence of carbonates, and pectins. The method may also be used for C in organic substances after wet oxidation, carbonates in minerals, CO_2 in beer, etc., sulphides and H_2S, nitrates by Devarda's alloy, H_2O by Fischer's method, halogens and their oxy-compounds, cyanides and their complexes, and oxalates.

D. R. GLASSON

2255. **A modified form of column chromatography.** K. Schlögel and A. Siegel (*Z. Naturforsch.*, 1954, **9b** [8], 570–571).—A longitudinally split column is employed. It is made of two glass troughs with perfectly ground edges, 50 cm long, 3·5 cm in internal diameter and walls 0·4 cm thick. The column is held together by steel bands and clamps. This form of column facilitates removal of adsorbent and recovery of fractions, and in many methods renders unnecessary the use of large volumes of eluent fluid and the fraction collector.

E. KAWERAU

2256. **Rotating-disc paper chromatography.** G. Caronna (*Chim. e Ind.*, 1955, **37** [2], 113–114).—By using a rotating disc, chromatograms can be obtained in a much shorter time than with stationary discs. Suitable apparatus is briefly described.

C. A. FINCH

2257. **Apparatus for automatically changing solvent polarity during chromatography.** R. R. Allen and D. N. Eggenberger (*Anal. Chem.*, 1955, **27** [3], 476).—A constant-pressure apparatus is described which adds components of a two-component mobile phase in the correct proportions. In this way the content of a developing solvent in partition chromatography can be changed progressively.

J. H. WATON

2258. **The determination of iodide - iodate activity in sodium radio-iodide (^{131}I) by automatic scanning of paper chromatograms.** J. J. Pinajian and J. E. Christian (*J. Amer. Pharm. Ass., Sci. Ed.*, 1955, **44**

[2], 107–109).—An automatic scanning and recording device is described for ^{131}I paper chromatography. The relationship of chart-drive speed and optimal slit width to the time constant of the counting-rate meter is discussed and the minimal slit width for the instrument used calculated. Integration of the curves gave areas representing the total activity at each spot. The ratio of the iodide - iodate activities thus determined differed from the relative readings of the counting-rate meter. R_F values of chromatograms of different width, using 75 per cent. methanol in an ascending system, are presented for I′ and IO_3'. The I′ spot is developed by spraying with starch - H_2O_2 solution and the IO_3' spot with ascorbic acid - starch solution. The identity of the spots is determined by the use of carrier iodide and iodate. N. M. WALLER

2259. **Simple colorimetric method for calibration of pipettes.** L. D. Ellerbrook (*Amer. J. Clin. Path.*, 1954, **24** [7], 868–874).—A standard coloured soln. of potassium dichromate or blood is prepared by making a known large dilution of the stock coloured soln. with accurate equipment. The pipette to be calibrated is used to make a similar dilution. The vol. delivered by the pipette being calibrated is determined by the ratio of the optical densities of the standard and unknown coloured soln.
R. S. TONKS

2260. **High rate of shear rotational viscometer.** E. M. Barber, J. R. Muenger and F. J. Villforth, jun. (*Anal. Chem.*, 1955, **27** [3], 425–429).—The design is given of a rotational viscometer for studying the behaviour of non-Newtonian liquids up to a shear rate of 10^6 reciprocal sec. The heating effect due to the high rate of shear is controlled by employing thin films of the test liquid, and by arranging for an equal heat path through the inner and outer cylinders, which are maintained at the same temp. by a temp.-control liquid. Data are given for several mineral oil - polymer blends. Good agreement is found with data already published for two of these mixtures, where different apparatus and techniques were employed. No permanent viscosity change is detected after prolonged and high rates of shear. The viscosity - rate of shear coefficient appears to be independent of temp. for the oils tested. J. H. WATON

2261. **The viscosity of normal and pathological human synovial fluids.** J. P. Johnston (*Biochem. J.*, 1955, **59** [4], 633–637).—A simple variable-velocity-gradient viscometer of low fluid capacity (0·3 ml) is described. Viscosity is measured by timing the rate of flow of fluid in a horizontal capillary, variation in the rate of flow being obtained by the application of various pressures. The errors involved in the use of capillary flow appear to be negligible.
J. N. ASHLEY

2262. **Simple apparatus for concentrating biological fluids of low protein content.** C. H. Grogan and E. Roboz (*J. Lab. Clin. Med.*, 1955, **45** [3], 495–498).—Details are given for the construction of a simple Cellophane-membrane dialysis apparatus in which dilute protein solutions can be concentrated 25- to 100-fold for subsequent paper electrophoresis. The protein solutions (10 to 20 ml of urine, cerebrospinal fluid, etc.) are dialysed at 5° C for up to 20 hr. against 15 per cent. aq. dextran or aq. poly(vinylpyrrolidone), which is stirred continuously during dialysis. Traces of the polymers which pass into the protein solutions do not interfere with the electrophoresis. W. H. C. SHAW

2263. **Density hydrometers for use in milk.** British Standards Institution (B.S. 734: 1955, 52 pp.).—Standards for density hydrometers for use in milk published in B.S. 734: 1937 (*Analyst*, 1937, **62**, 613) are revised. Three sizes of hydrometer are provided for use with varying quantities of liquid (200 ml, 130 ml and 57 ml). Two hydrometers in each size cover the density ranges 1·025 g per ml to 1·035 g per ml (normal) and 1·015 g per ml to 1·025 g per ml (low density) based on density measured at 20° C in a liquid having surface tension 46 dynes per cm. Corresponding hydrometer jars are specified. Appendices cover: (1) method of use, (2) scale-error corrections, (3) correction of hydrometer reading at other temp. to density at 20° C, (4) tables giving non-fatty solids corresponding to fat content between 1·00 per cent. and 10·00 per cent. and densities in the range of the hydrometers, (5) corrections to give density of milk at temp. other than 20° C, (6) application of hydrometer readings to determination of wt. and vol. of bulk milk.
D. G. FORBES

2264. **Centrifuge tubes and sedimentation vessels for the determination of visible dirt in milk.** British Standards Institution (B.S. 736: 1955, 11 pp.).—Standards published in 1937 (see *Analyst*, 1937, **62**, 308) for centrifuge tubes and sedimentation vessels are revised. *Part* 1 specifies three sizes of centrifuge tube with total capacity of calibrated portion as follows: Size 1.—0·02 ml; Size 2.—0·05 ml; Size 3.—0·2 ml. The internal diameter has been increased from $10{\cdot}5 \pm 0{\cdot}3$ mm to $12{\cdot}0 \pm 0{\cdot}3$ mm. *Part* 2 details standards for sedimentation vessels having a shorter tapered portion. D. G. FORBES

See also Abstracts 2047, 2149.

Optical

2265. **Applications of curved-crystal X-ray spectrometers. Micro-analysis and simultaneous analysis.** L. S. Birks and E. J. Brooks (*Anal. Chem.*, 1955, **27** [3], 437–440).—The use of a curved reflection-type focusing crystal in a fluorescent X-ray spectrometer permits both macro and micro amounts of material to be analysed. The results from a 1-mg sample with a curved-crystal spectrometer are as good as those for samples of several grams employing a flat-crystal spectrometer. The technique is applied to the determination of p.p.m. of Nb, Hf, Ta, Th and U in Fe, which conventional emission spectroscopy cannot handle. With Tl as an internal standard, the results show a standard deviation of $\simeq \pm 13$ per cent. of the amount present. Simultaneous analysis for several elements may be carried out, one curved crystal and a detector being employed for each element. The absence of any need for collimators considerably simplifies the apparatus. The simultaneous analysis for Cr, Ni and Mn in steel is described, but the determination of more than three elements is possible. J. H. WATON

2266. **Rotatory dispersions of some steroids, amino acids and peptides, using a new spectropolarimeter.** E. Brand, E. Washburn, B. F. Erlanger, E. Ellenbogen, J. Daniel, F. Lippmann and M. Schen (*J. Amer. Chem. Soc.*, 1954, **76** [10], 5037–5040).—A new photo-electric spectrophotometer is described for rotatory dispersion studies for identification and as criteria of purity of steroids, amino acids and peptides. The light from a Western Union K-100 zirconium arc lamp in quartz envelope, that is interchangeable with a General Electric Na-1 sodium lamp or a Hanovia 16A13

mercury quartz burner, is passed through a Beckman DU monochromator into a polarimeter. The monochromator has a special base and accessories to facilitate proper alignment of light source, monochromator and polarimeter, which has quartz Rochon polariser and analyser prisms and an analyser circle reading to 0·001°. No Lippich prism is used. Light intensity is measured by a photo-electric attachment consisting of RCA1P21 and 1P28 photomultiplier tubes and a Photovolt photometer with a special scale. Optical rotations are measured by the method of symmetrical angles. The standard deviation of each reading was found to vary from ± 0·001° to ± 0·004° depending on the wavelength of the incident light. N. E.

2267. **Photo-electric Raman spectrometer.** J. U. White, N. L. Alpert and A. G. Debell (*J. Opt. Soc. Amer.*, 1955, **45** [3], 154–166).—The instrument described has a 1200-line per mm replica grating and can record a complete Raman spectrum of a 5-ml liquid sample in six minutes, if rapid survey work is being undertaken. For highest accuracy, scanning time is about 1 hr. B. S. Cooper

2268. **Weighing-pipette method for preparing infra-red gas standards for ether and alcohol.** F. Pristera and A. Castelli (*Anal. Chem.*, 1955, **27** [3], 457–459).—A method is described for the preparation of infra-red gas standards of ether and ethanol by using a micro weighing-pipette. The filled pipette is introduced into the threaded opening of a metal gas-cell and is broken, inside the cell, by touching with a thin metal rod heated to ≃ 50° C. The rod is withdrawn and the cell opening is suitably closed, infra-red measurement being made after 30 min. to allow complete vaporisation of the ethanol or ether. Good results were obtained from synthetic standard mixtures of ether and ethanol in air. G. P. Cook

2269. **New infra-red microcell.** W. H. T. Davison (*J. Opt. Soc. Amer.*, 1955, **45** [3], 227).—Two simple forms of cell are described in which the liquid is held in a defined area by capillary attraction. A cell consists of a pair of rock-salt plates clamped together, one plate being plane whilst the other has a milled channel cut in it with an engraving machine. Typical examples quoted give sample thicknesses in the range 0·001 to 0·01 cm with corresponding liquid vol. of 0·2 to 6·2 μl. B. S. Cooper

2270. **Recording vacuum infra-red prism-grating spectrometer.** K. P. Yates and R. F. Buhl (*J. Opt. Soc. Amer.*, 1955, **45** [3], 192–201).—The evacuable infra-red spectrometer described ' includes a potassium bromide prism and three gratings (7500, 1800 and 1000 lines per inch, respectively), the appropriate component being selected according to the desired resolution and wavelength range. The total wavelength range available is 1 to 20 μ. A new form of carbon rod infra-red source, which operates at about 1500° C, is also described. B. S. Cooper

2271. **Gas detectors and analysers.** Infra Red Development Co., Ltd. (Inventor: W. B. Bartley) (Brit. Pat. 727,600, Date Appl. 22.8.52).—The calibration curve of infra-red gas detectors and analysers is found to vary, partly owing to variations in temp. and partly to variations in pressure resulting from surface chemical action in the detector chamber. The pressure variations can be eliminated by means of a collapsible reservoir (metal bellows) in communication with a detector chamber through a capillary passage. The volume of the bellows can be contracted by a screw bearing on the dome. J. M. Jacobs

2272. **Instrumental variability of a Model 7 Coleman photo-nephelometer.** H. J. Keily and L. B. Rogers (*Anal. Chem.*, 1955, **27** [3], 459–461).—Inaccuracies encountered in the use of the Coleman photo-nephelometer are due to a downward drift of the readings and also to the variability of the distilled water used as a blank. An alternative procedure is described, using a two-standard method, and hence a hypothetical blank, with repeated adjustment of the instrument before each reading. At the normal sensitivity of the instrument, a standard deviation for individual measurements of 0·28 of a (Nephelos) unit is obtained. Although the standards supplied by the manufacturers differ from their nominal values, their stability is very satisfactory. J. H. Waton

2273. **Simple adaptation of the Beckman DU spectrophotometer as a spectrofluorimeter.** A. G. Gornall and H. Kalant (*Anal. Chem.*, 1955, **27** [3], 474–475).—The Beckman DU spectrophotometer can be used as a spectrofluorimeter by adapting with a few simply made parts the fluorescence accessory provided with the instrument. The apparatus is calibrated with quinine in 0·1 *N* H_2SO_4, the light from a 500-watt tungsten lamp being passed through a Corning 3-mm 9863 filter. With the photomultiplier at max. sensitivity and the sulphuric acid blank at zero, a concn. of 0·15 μg of quinine sulphate per ml of soln. gives a scale reading of 100 at a slit width of ≃ 1·8 mm. For lower concn. a straight-line relationship holds. J. H. Waton

2274. **Method of continuous refractometry in chromatographic frontal analysis.** S. S. Salazkina, L. T. Solov'ev and V. A. Yurev (*Vopr. Med. Khim.*, 1953, **6**, 175–179; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 41,790).—A device is described which enables continuous refractive-index measurements to be made on a liquid flowing from an absorption column. Examples are given showing the use of the device in a study of the behaviour of ionites in amino-acid separations. E. Hayes

2275. **Flame-photometric method of determining calcium in solution.** D. N. Ivanov (*Zh. Anal. Khim., SSSR*, 1954, **9** [6], 344–353).—The use of an interference filter with max. transmission at 620 mμ permits the emission from Ca to be separated from that of K and of Na, when these elements are present in the solution being fed into an acetylene - air flame. Ca is determined by means of a selenium photo-element. The apparatus is illustrated. G. S. Smith

2276. **Tolansky gauge for rapid measurement of film thickness.** T. M. Green and L. N. Hadley (*J. Opt. Soc. Amer.*, 1955, **45** [3], 228–229).—In place of a travelling microscope to measure fringe spacing and fringe shift, the arrangement described makes use of the displacement produced when a plane slab of glass is set obliquely in the optical path and altered in inclination to the light beam. B. S. Cooper

2277. **Improved solution light-scattering cell.** F. P. Price and B. H. Zimm (*J. Opt. Soc. Amer.*, 1955, **45** [3], 229).—A composite glass and plastic cell assembly is described which enables scattered light to be measured over a wider range of angles than with rectangular or cylindrical cells. It is

also stated that the background light is very low at all accessible angles. B. S. COOPER

2278. **Further investigations in the spectro-isotopic assay technique for lithium.** G. K. Werner, D. D. Smith, S. J. Ovenshine, O. B. Rudolph and J. R. McNally, jun. (*J. Opt. Soc. Amer.*, 1955, **45** [3], 202–205).—A high-resolution optical system has been devised that is suitable for photo-electric detectors to be applied to the measurement of the intensities of the isotope components of the Li line 6707 A, which are separated by 0·16 A. When used in conjunction with an improved type of hollow cathode source, ^{6}Li may be determined with a precision of $\pm$ 0·46 per cent. at the 30 per cent. isotope abundance level. The equipment has been tested and found to be generally satisfactory over the range 0·2 per cent. to 98·3 per cent. of ^{6}Li. B. S. COOPER

Thermal

2279. **Air stirring for bomb calorimeter.** J. W. Whitaker, A. K. Ghosh and R. N. Chakravorty (*J. Sci. Ind. Res. India, B*, 1955, **14** [1], 24–26).—The advantages of stirring the contents of a bomb calorimeter by bubbling air instead of using mechanical stirring are described. R. J. COLE

2280. **Non-bumping digestion heater.** A. Steinbergs (*Anal. Chem.*, 1955, **27** [3], 472).—A non-bumping digestion heater suitable for Kjeldahl flasks is described. The heating is provided by a Nichrome wire element, which is wound round the inside of a fireclay support, directing the heat towards the sides of the enclosed flask. J. H. WATON

2281. **Automatic micromuffle for determination of ash in carbonaceous material.** R. Meyrowitz and C. J. Massoni (*Anal. Chem.*, 1955, **27** [3], 475–476).—A micromuffle is described in which the furnace drive and moving furnace are parts that are commercially available (in the U.S.A.). J. H. WATON

2282. **An improved apparatus for the determination of gaseous elements in metals by vacuum fusion on a micro scale.** J. N. Gregory and D. Mapper (*Analyst*, 1955, **80**, 225–230).—A vacuum fusion apparatus is described and illustrated which presents some improvements on an earlier apparatus (Gregory *et al.*, *Brit. Abstr. C*, 1953, 420). The improvements relate mainly to more efficient pumping, recovery of the evolved gases and the introduction of a low-pressure method of gas analysis. The last improvement enables the time for the examination of a series of samples to be reduced to about half a day compared with 2 or 3 days by the previous method. A. O. JONES

Electrical

2283. **Output control of the Honeywell - Brown potentiometer in recording variable currents, with regard to polarographic diffusion currents.** P. Papoff and I. M. Vezzosi (*Ric. Sci.*, 1955, **25**, 302–313).—By using an electrical circuit, with a Honeywell - Brown Electronik recording potentiometer, capable of reproducing the current - time curve corresponding to the life of a drop from the polarograph electrode, measurements are carried out to determine the dependence between max. efficient current and the registered max. height, with respect to the average height. Measurements are taken for a drip period interval from 1·2 to 18 sec. with various damping values. Many data are tabulated. C. A. FINCH

2284. **Glass electrodes.** British Standards Institution (B.S. 2586: 1955, 16 pp.)—Standards are detailed for construction and dimensions of ordinary and micro-electrodes of three types: (*i*) general purpose (pH 1 to 10), (*ii*) high pH (pH 9 to 13), (*iii*) wide pH range (pH 1 to 13), having in the bulb a recommended reference electrode of the Ag|AgCl|HCl type. Conditions are laid down for potentiometric determination of electromotive efficiency and of d.c. resistance of the electrodes; means of identification and precautions for packing are suggested. D. G. FORBES

2285. **Salt bridges of porous glass and ion-exchange membranes.** W. N. Carson, jun., C. E. Michelson and K. Koyama (*Anal. Chem.*, 1955, **27** [3], 472–473).—Salt bridges made from porous glass and from ion-exchange membranes last longer than conventional bridges, and show little change in characteristics over several months provided that they are not allowed to become dry. They are superior mechanically and have a considerably lower resistance. Bridges of porous glass are attacked by F′ and by caustic alkali, and show adsorption of dyestuffs. When stored in distilled water, severe leaching of the salt occurs. Ion-exchange membranes do not give low junction potentials, but have the advantage that they do not allow any soln. to pass and can thus prevent unwanted ions from passing from one half-cell to the other. The construction of examples of both types of bridge is described. J. H. WATON

2286. **Electrometric titrations in anhydrous acetic acid.** G. Jander and H. Klaus (*J. Inorg. and Nuclear Chem.*, 1955, **1** [1–2], 126–142).—Neutralisation reactions in anhydrous acetic acid are followed potentiometrically by using a special type of non-aqueous reference "damped" electrode. A metal electrode is set in a capillary forming the inner compartment of a concn. cell. Since the capillary is just small enough to prevent appreciable diffusion, changes in potential occur at the electrode in the outer compartment only when the liquid there is titrated. The apparatus is apparently not restricted in its application to glacial acetic acid solutions. The most satisfactory electrode material is gold wire or foil, which is usable over the whole range of H· and Ac′ concn. and yields reproducible potentials. Solutions of alkali acetates are titrated with 0·25 *N* $HClO_4$, their basic strengths increasing with increasing cationic radius, from Li to Na to K. Organic bases, *e.g.*, aniline, dimethylaniline, diethylaniline, 1-naphthylamine and pyridine are similarly titratable with 0·25 *N* or 0·5 *N* $HClO_4$, the limiting concn. being 0·01 *N*. Large changes in potential at the end-point occur when the salts formed are only slightly sol. in acetic acid. Titrations with H_2SO_4 give similar results. When sulphonic acids are used as titrants, toluenesulphonic acid monohydrate with the requisite amount of acetic anhydride is specially recommended. Conductimetric results are presented for benzenesulphonic acid and HBF_4 titrated with diethylaniline. Best results are obtained by using $HClO_4$ as standardising acid. The concept that $HClO_4$ and H_2SO_4 in glacial acetic acid solutions have a super-acid character is refuted by comparing the forward and reverse titrations of amines; increase in the basic strength of the amines, compared with that in aq. media, renders them titratable in anhydrous acetic acid. D. R. GLASSON

2287. **Titrations with a high-frequency titrimeter of resistance type. (Variation of the shape of the titration curve with concentration.)** K. Nakano (*J. Chem. Soc. Japan, Pure Chem. Sect.*, 1954, **75** [5], 494–498).—By using a high-frequency titrimeter ($\simeq$ 1 Mc.p.s.) designed by the author (*Anal. Abstr.*, 1954, **1**, 2031), the shape of neutralisation titration curves with different concn. is studied precisely. When the change of resistance of the circuit is plotted against the molar ratio of the reactants, the curve shows a max. at the equiv. point for a high concn. of reactants ($> 0{\cdot}2\ N$), and a min. for a low concn. ($< 0{\cdot}01\ N$). For intermediate concn., the equiv. point is obscure. Strong bases behave in a similar way to one another, as do strong acids. When a weak acid or base participates in the reaction, the curve shows different shapes. The reason for these differences is discussed in relation to the resistance - concn. diagram of the titrimeter (*Cf. Anal. Abstr.*, 1955, **2**, 1730).

K. Saito

2288. **Improved apparatus for zone electrophoresis.** A. M. Crestfield and F. W. Allen (*Anal. Chem.*, 1955, **27** [3], 422–423).—An improved apparatus for electrophoresis on filter-paper is described. It incorporates a sprinkler system which cools the underside of the plate supporting the filter-paper sheet, preventing the evaporation of the electrolyte and permitting as much as 0·10 to 0·25 watts per sq. cm to be applied. The same system sprays hot water at the end of the electrophoresis, so that the paper is dried within 10 min., and zone movement prevented. Mobilities of charged material may be determined with caffeine as an uncharged reference material, whose presence is readily detected by u.v. light. J. H. Waton

2289. **Apparatus and technique for two-dimensional paper ionophoresis.** T. H. Mead (*Biochem. J.*, 1955, **59** [4], 534–543).—A new apparatus is described for two-dimensional paper ionophoresis, in which the paper is cooled efficiently to minimise evaporation and the pH of the buffer solution in the paper is changed, after ionophoresis in one direction, by exposure to the vapour from a solution with a suitable and substantially constant partial pressure of NH_3. This is accomplished by ionophoresis in one direction in acid buffer (phthalate) containing a weak acid (boric acid) capable of forming, with NH_3, a buffer effective at the pH desired for ionophoresis in the second direction. A method is suggested for the determination of the flow of electrolyte at different points on the paper. The new apparatus qual. separates glutamic and aspartic acids, cystine, glycine, alanine, valine, leucine, phenylalanine, serine, threonine, tyrosine, hydroxyproline, proline, histidine, lysine and arginine, but does not separate leucine from *iso*leucine. The developed ionophoretogram is ready within 9 hr. of starting an experiment. The scope of the method is discussed. J. N. Ashley

2290. **Methanometers.** National Coal Board (Inventor: F. W. Pritchard) (Brit. Pat. 727,461, Date Appl. 14.12.50).—The methanometer consists of two air-tight chambers each containing an electrically heated platinum filament. The first filament, acting as a dummy for comparison purposes, is positioned in a chamber containing air. A stream of the gas to be analysed is passed through the second chamber, in which the filament is arranged co-axially within a thin cylindrical metal sheath having an internal diameter of < 5 mm. The change in resistance of the second filament, due to its temp. change in the presence of the air - methane mixture, is determined by means of a Wheatstone bridge circuit. J. M. Jacobs

2291. **Means for analysing gaseous substances [particularly for detecting methane in coal mines].** J. J. D. Walton (Brit. Pat. 727,891, Date Appl. 4.5.51).—The device is based on the changes in the sound-propagation characteristics of a gas brought about by changes in its composition. A self-excited oscillator supplies electrical energy to a sending transducer adapted to transmit sound energy through a vessel containing a variant gas. A receiving transducer receives the sound energy. The electrical signals obtained are compared for phase and amplitude by transmission of sound energy through a datum gas in a vessel.

J. M. Jacobs

2292. **A capacity-change drop counter.** R. E. Ricketts (*Analyst*, 1955, **80**, 213–214).—The circuit diagram is given for a simply constructed and reliable capacity-change drop counter. Fractions of 1 to 5 ml may be automatically collected. The apparatus is designed for the collection of a large number of fractions of eluate from resin columns used for the fractionation of protein hydrolysates. It operates in conjunction with a chromatographic table (Hough *et al.*, *J. Chem. Soc.*, 1949, 2511), a set number of drops being counted into separate test-tubes. A. O. Jones

2293. **A new apparatus for the automatic measurement of radioactivity.** W. Bolliger and G. G. Poretti (*Experientia*, 1955, **11** [3], 115–116).—An apparatus is described which can measure 31 radioactive samples automatically. The time required for collecting a preset number of counts is also automatically registered by 31 mechanical registers. Absorption measurements are also possible.

R. S. Tonks

2294. **Mass spectrometer.** Bendix Aviation Corp. (Brit. Pat. 727,683, Date Appl. 12.8.52; U.S.A. 2.10.51).—A time-of-flight mass spectrometer is provided with two ion-accelerating grids, and a predetermined voltage relation is employed between the grids and the backing plate. By this means, the initial position error (space spread) and the error due to the initial velocity of the ions are eliminated.

J. M. Jacobs

Errata.—May (1955) issue, abstract 1357, line 2.
The abbreviation of the journal should be *Meded. LandbHogesch. Gent.*

July (1955) issue, abstract 1816, line 15.
For cyanate *read* hydroxycyanide.

Translations

The following papers of interest to analytical chemists have been translated into English.

CONSULTANTS BUREAU

Copies of these papers can be obtained from Consultants Bureau, 152 West 42nd Street, New York 18, N.Y., U.S.A. Each translation costs $7.50 and orders should state title, author(s) and English page number. The English page number is given in parentheses after the Russian page number.

These translations can also be seen in the library of the Chemical Society, Burlington House, London, W.1.

***Bull. Acad. Sci., U.S.S.R.*—**

Determination of the individual hydrocarbon composition of gasolines by the combination method. II. Two gasolines from Kazanbulak petroleums—B. A. Kazansky, A. F. Plate, E. A. Mikhailova, A. L. Liberman, M. I. Batuev, T. F. Bulanova and G. A. Tarasova, 1954, [3–4], 266 (215).

Determination of the individual hydrocarbon composition of gasolines by the combination method. III. Surakhan gasolines—B. A. Kazansky, G. S. Landsberg, A. F. Plate, A. L. Liberman, E. A. Mikhailova, P. A. Bazhulin, M. I. Batuev, S. A. Ukholin, T. F. Bulanova and G. A. Tarasova, 1954, [3–4], 278 (225).

Determination of the individual hydrocarbon composition of gasolines by the combination method. IV. Gasoline from Tuimazin petroleum—B. A. Kazansky, G. S. Landsberg, A. F. Plate, P. A. Bazhulin, E. A. Mikhailova, A. L. Liberman, M. M. Sushchinsky, G. A. Tarasova, S. A. Ukholin and S. V. Voronko, 1954, [5–6], 456 (387).

***J. Gen. Chem., U.S.S.R.*—**

Isolation of β-sitosterol from crude phytosterol and its analysis—A. M. Khaletsky and I. M. Yurist, 1954, [3], 535 (549).

***J. Anal. Chem., U.S.S.R.*—**

A horizontal a.c. arc as spectrum excitation source for ores and minerals—A. K. Rusanov and V. M. Alekseeva, 1954, **9**, 183 (203).

Ultra-violet colorimetric determination of small amounts of ketones—S. A. Shchukarev, S. N. Andreev and O. V. Sapozhnikova, 1954, **9**, 193 (213).

Determination of beryllium in bronze by means of a cation-exchange resin—D. I. Ryabchikov and V. E. Bukhtiarov, 1954, **9**, 196 (217).

Organic co-precipitants. I. The theoretical basis of organic co-precipitant action—V. I. Kuznetsov, 1954, **9**, 199 (221).

Qualitative analysis of organosilicon compounds by infra-red absorption spectroscopy—A. P. Kreshkov Yu. Ya. Mikhailenko and G. F. Yakimovich, 1954, **9**, 208 (231).

Colour reactions of carbazole, indole, pyrrole and some of their derivatives with bromonitroindanedione—G. Vanag, 1954, **9**, 217 (241).

Colour reactions of certain mercuriated aromatic amines with nitrites—I. M. Korenman and A. A. Belyakov, 1954, **9**, 220 (245).

Iodimetric determination of cadmium—V. I. Kumov, 1954, **9**, 229 (255).

Iodimetric determination of arsenic acid using organic solvents—G. B. Shakhtakhtinsky, 1954, **9**, 233 (259).

Quantitative determination of sodium as antimonate—K. S. Cheshev, 1954, **9**, 239 (265).

The application of mathematical statistics in analytical chemistry—S. A. Gusanskaya, 1954, **9**, 245 (273).

DEPARTMENT OF SCIENTIFIC AND INDUSTRIAL RESEARCH

The following translations can be obtained from D.S.I.R. Technical Information and Documents Unit, Cunard Building, 15, Regent Street, London, S.W.1, at the prices shown.

***Bull. Acad. Sci., U.S.S.R.*—**

An investigation of the conditions of separation and gravimetric determination of silicic acid. I.—E. N. Egorova, 1953, [3], 419. Price 60s.

***Biochemistry*—**

A method for the determination of flavone substance in plants—A. R. Guseva and M. N. Nestyuk, 1953, **18** [4], 480. Price 32s.

SCIENCE MUSEUM LIBRARY

Photo-copies of the following translations can be obtained from the Science Museum Library, South Kensington, London, S.W.7.

***Comp. Rend. Acad. Sci., U.S.S.R.*—**

Spectrophotometric methods for determining the intensity, form and width of infra-red absorption bands of liquids—A. V. Ioghansen, 1953, **92**, 919.

The preparation and use of ion-exchange membranes for electrodialysis—O. N. Ghrīghorov, K. F. Kulīkova and A. I. Sharapova, 1954, **94**, 501.

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use in the text of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångstrom unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher -e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
critical	crit.
crystalline	cryst.
crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c.p.s.
decompos -ing, -ition	(decomp.)
density	ρ
density, relative	d or wt. per ml
derivative	deriv.
dilute	dil.
direct current	d.c.
distilled	dist.
electromotive force	e.m.f.
electron-volt	eV
equivalent	equiv.
experiment	expt.
foot, feet	ft.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
horse-power	h.p.
hour	hr.
hydrogen ion concentration	$[H^{\cdot}]$
hydrogen ion exponent	pH
inch	in.
infra-red	i.r.
insoluble	insol.
kilogram	kg
kilovolt	kV
kilowatt	kW
maxim -um, -a	max.
melting-point	m.p.
microcurie	μC
microgram	μg
microlitre	μl
micron	μ
milliampere	mA
millicurie	mC
milligram	mg
millilitre	ml
millimetre	mm
millimicron	mμ
millivolt	mV
minimum	min.
minute (time)	min.
molar (concentration)	M
molecul -e, -ar	mol.
normal (concentration)	N
number	no.
observed	(obs.)
ounce	oz
part	pt.
patent	pat.
parts per million	p.p.m.
per cent. wt. in wt.	per cent. w/w
per cent. wt. in vol.	per cent. w/v
per cent. vol. in vol.	per cent. v/v
potential difference	p.d.
pound	lb
precipitate	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ
relative humidity	R.H.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda$
square centimetre	sq. cm
standard temperature and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition the following symbols are used—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P., or D.A.B., together with the identifying numeral.

Radicles are represented by the usual symbols; positive ions have superscript dots and negative ions superscript dashes, *e.g.*, $Cu^{\cdot\cdot}$, $Al^{\cdot\cdot\cdot}$, Cl', SO_4''. Metals that exist in more than one valency state are represented by their symbols with appropriate superscript roman numerals, *e.g.*, ferric iron becomes Fe^{III} and cuprous copper Cu^{I}.

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ANALYTICAL ABSTRACTS

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